FEBRUARY 1947

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VOL. 155 PART 2

Principal Contents

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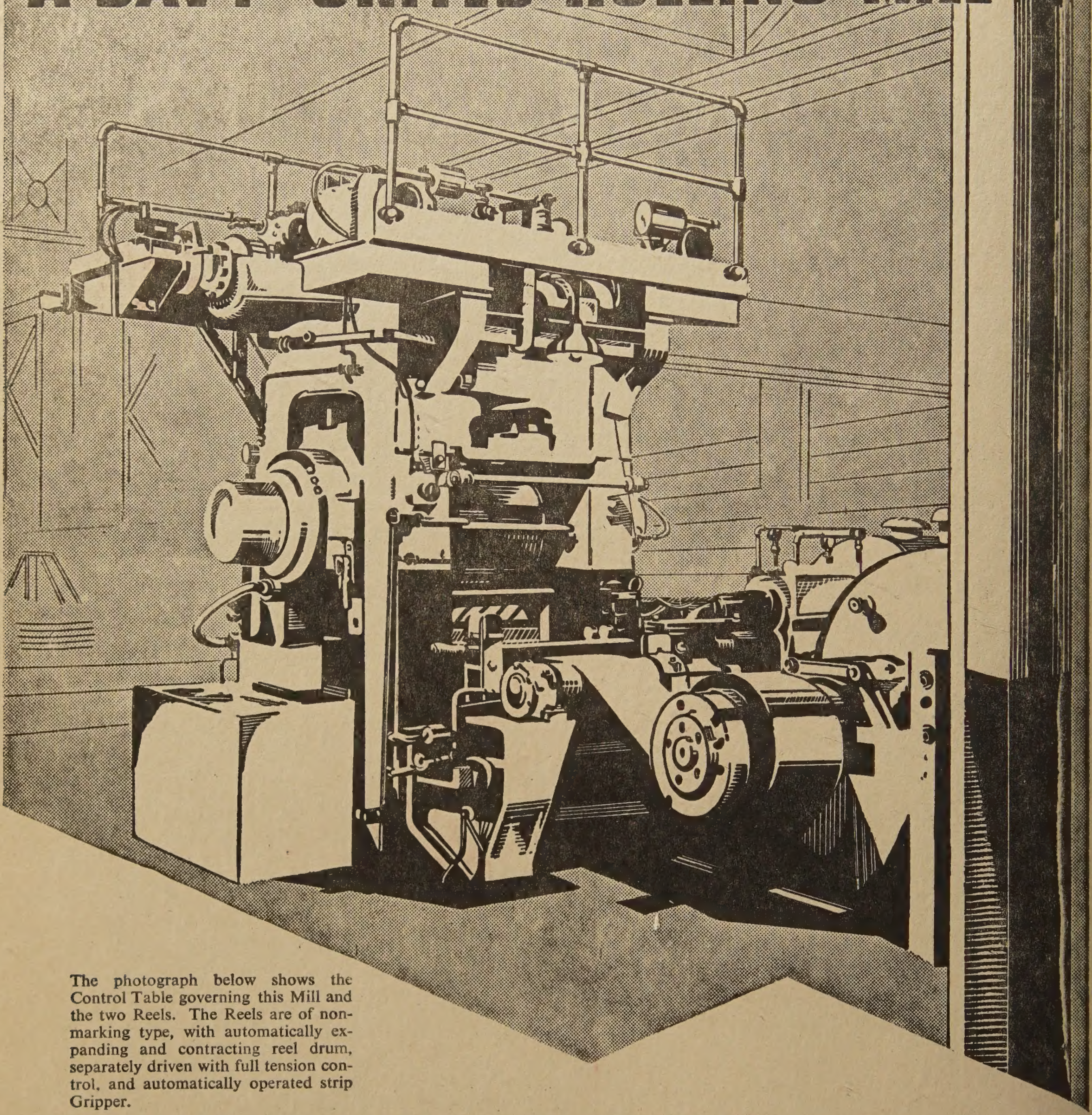

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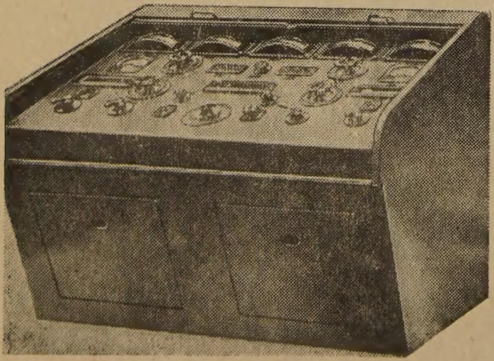

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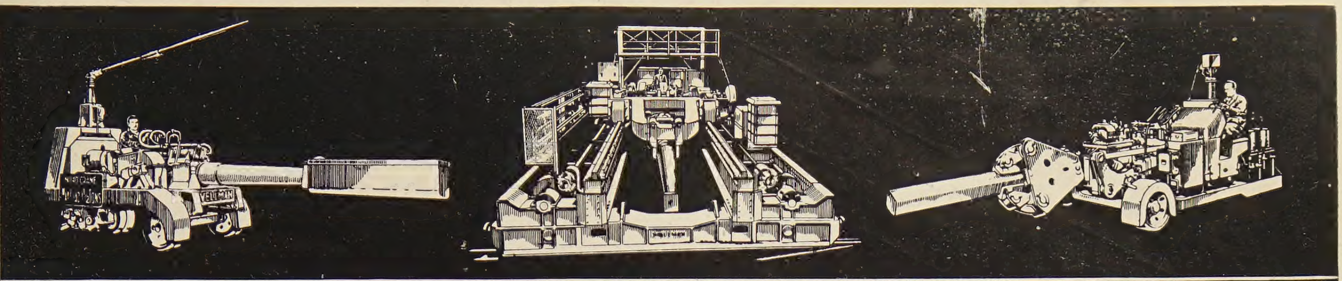

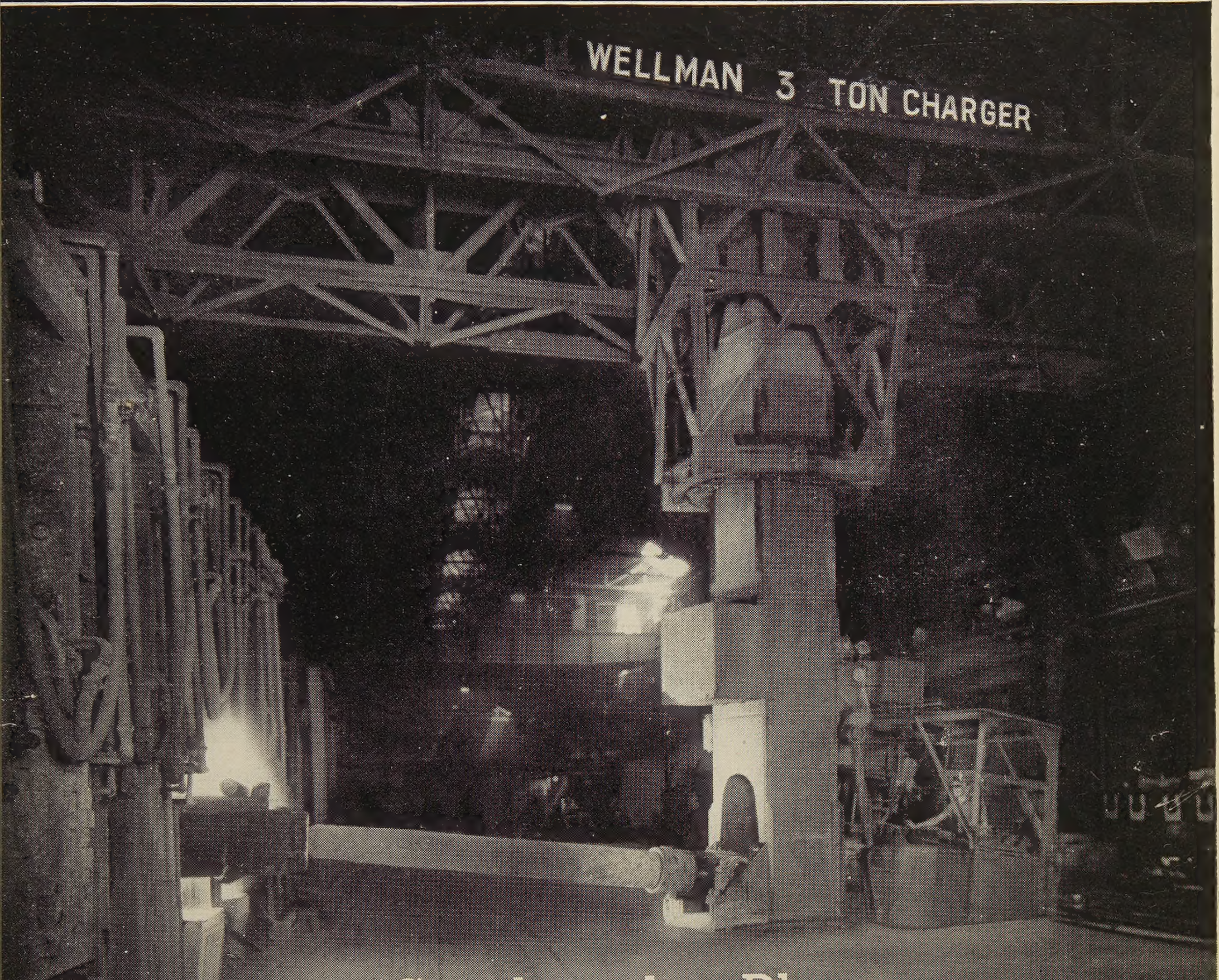
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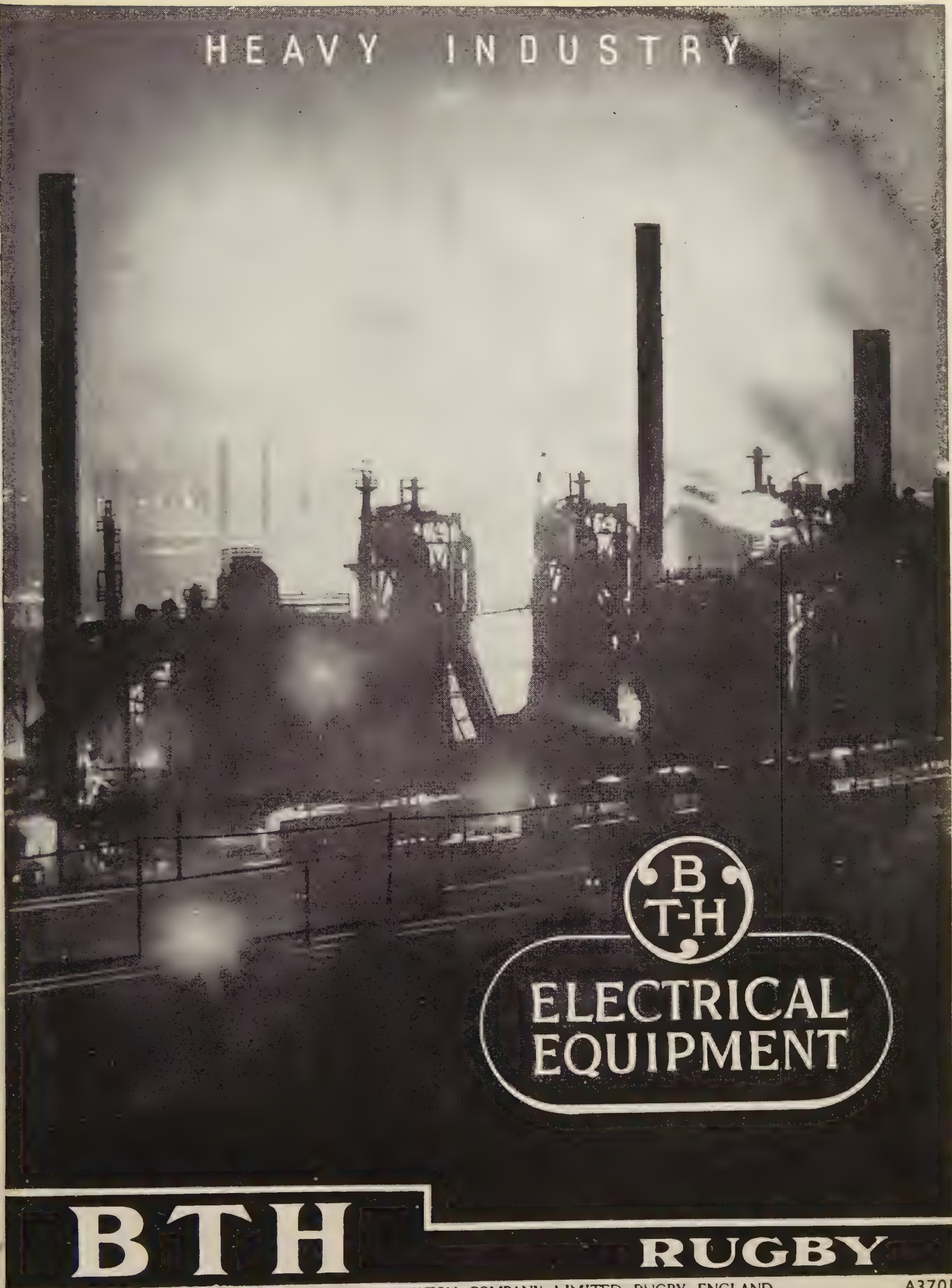

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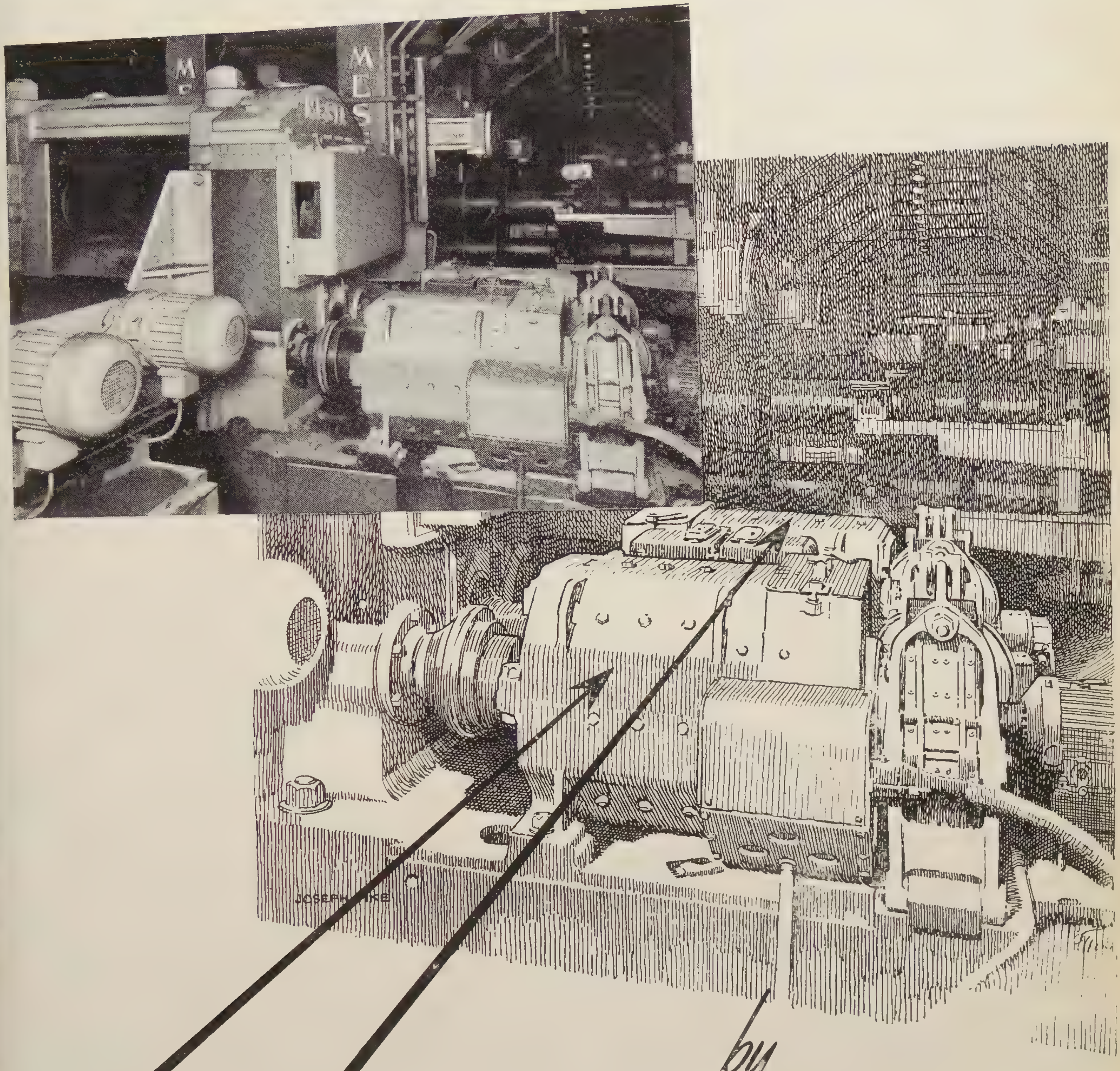

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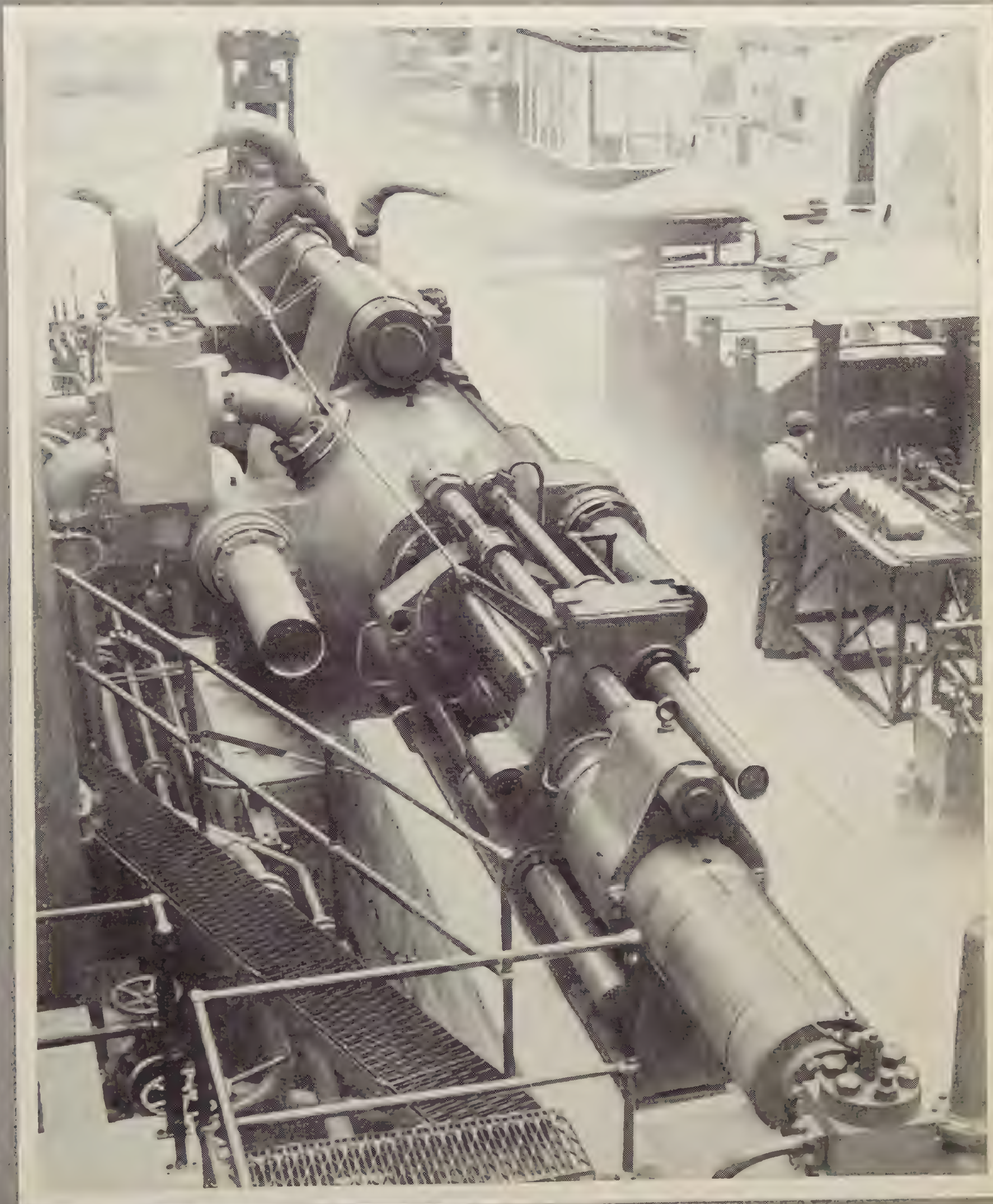

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CONTENTS

IRON AND STEEL INSTITUTE PAPERS

BRITISH IRON AND STEEL RESEARCH ASSOCIATION

IRON AND STEELWORKS ENGINEERING

NEWS

ABSTRACTS and BOOK REVIEWS

THE IRON AND STEEL INSTITUTE, 4 GROSVENOR GARDENS, LONDON, S.W.1

MR. BENJAMIN TALBOT

President 1928

CHAIRMAN and Managing Director of the South Durham Steel and Iron Co., Ltd., and of Cargo Fleet Iron Co., Ltd., Mr. Benjamin Talbot has been closely connected with all aspects of iron and steel manufacture for more than half a century. During this period he has not only been a witness to the advances made by the industry in various directions, but has made his own very substantial contributions to progress in steelmaking technique.

He served his apprenticeship in steelmaking at the Ebbw Vale Works, and went to the United States in 1890 to become Superintendent of the Southern Iron and Steel Company of Chattanooga, Tennessee, where he inaugurated the basic open-hearth process. He carried out a great deal of experimental and developmental work at the Tennessee plant, which was operating on a high silicon pig-iron. This led him to develop a preliminary refining operation designed to reduce the silicon content, and that item of plant has now become well known as the basic-lined gas-heated mixer.

In 1893 he became Steelworks Superintendent at the Pencoyd Steelworks, Pennsylvania, where he commenced the manufacture of basic open-hearth steel. Six years later he began the continuous steelmaking process which now bears his name, and in 1900 he presented his first paper to the Iron and Steel Institute on that subject.

The continuous steel process was developed further on Mr. Talbot's return to Great Britain in 1900. Some of the largest steel companies in this country, including the two with which he is now associated, introduced the continuous process and with his inventive capacity Mr. Talbot has for many years been engaged in improving the design of large steelmaking furnaces. Among his more recent inventions is the " Talbot Hydro-Carbon Lining Process " for the prevention of corrosion in cast iron and steel pipes.

During the First World War, Mr. Talbot was an unofficial adviser to the Ministry of Munitions, and in the period between the two wars his services were employed by various Government and official organisations.

In recognition of his inventions and researches in steelmaking, Mr. Talbot was awarded, in 1908, the Bessemer Medal of the Iron and Steel Institute, and the Elliott Gresson Gold Medal and the John Scott Medal of the Franklin Institute. He became President of the Iron and Steel Institute in 1928 and in the same year served as President of the National Federation of Iron and Steel Manufacturers.

Lafayette

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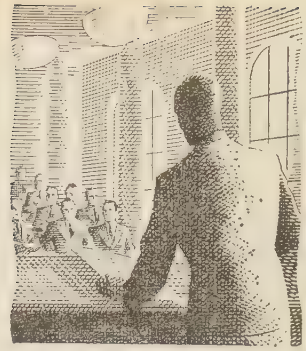

Application of Slag Control and an Investigation of Basic Open-Hearth Furnace Slags*

By T. Fairley Ph.D., M.Sc., F.Inst.P., A.F.R.Aë.S., F.I.M.†

SYNOPSIS

A method of slag control for fixed basic open-hearth furnaces which is based on a combination of existing practices is described, and the results are given of a statistical survey of the chemical compositions of the melting and tapping slags of 250 casts made by the application of this method. Graphs correlating the values of slag basicities as determined by different formulae are included.

INTRODUCTION

MUCH progress has been made during the last few years in the development of basic open-hearth furnace slag control. Various methods which have been used in the development of slag-control systems include the following: (*a*) Chemical analysis of the slag, (*b*) visual examination of slag pancakes, (*c*) colours of slag powders, and (*d*) microscopic examination.

(*a*) *Chemical Analysis of the Slag*

A very useful method, based on chemical analysis, has been described by Bacon.[1] Briefly, this method is as follows: Samples of slag and metal are taken about 30 min. before the bath is clear-melted, and quick methods of analysing the slag for SiO_2, P_2O_5, and Fe enable the required amounts of lime and oxide to be calculated by the time the bath is ready for such additions. Bacon (*loc. cit.*) gives detailed explanations of the calculations for the required feed based on the following considerations.

Since basic slag and limestone both contain approximately 50% of CaO, the weight of slag at melting is approximately equal to the weight of limestone put in the furnace with the charge. The percentages of SiO_2 and P_2O_5 in the melting slag are added together, and to this is added the amount of P_2O_5 which would be formed if the phosphorus in the metal at melting could be transferred, as P_2O_5, to the slag. If a maximum phosphorus content of 0·035% is required in the metal the $(SiO_2 + P_2O_5)$ in the tapping slag should not exceed 18%. Hence the amount of limestone which must be added after melting in order to reduce the " total acids " to 18% may easily be calculated. Allowance must be made for the amount of CaO which enters the slag from the dolomite of the banks during the refining period.

The amount of oxide required to remove 0·01% of carbon or phosphorus from 1 ton of metal and the amount required to raise the slag Fe of 1 ton of slag by 1% are known. Hence the amount of ore required to obtain the correct tapping analysis may be calculated. Allowance must be made for furnace oxidation.

* Received 20th June, 1946.

† The Park Gate Iron and Steel Co., Ltd., Rotherham.

Bacon,[1] Maurer,[2] Stengel,[3] and Knight [4] indicate that the slag may be analysed in approximately 30 min.

(b) *Visual Examination of Slag Pancakes*

The first American study of basic open-hearth slag by solidification tests to be published was by Janitzky [5] in 1929. A general correlation was made of the readily observed characteristics of similarly solidified basic open-hearth slag samples in respect of their chemical composition, colour, surface design, conformation, and texture. He emphasized that the desirable slag characteristics and composition must be determined for each plant, as local variations in the composition of the charge and fluxes would undoubtedly affect the coloration, surface design, and texture of the slag tests. Since then, various workers have described the relation between the lime/silica ratio of a slag and the appearance of a slag pancake. Such relationships have been described by Back,[6] Malloy,[7] Washburn and Philbrook,[8] Bischof,[9] Courtright,[10] Mauer,[11] and McKimm.[12]

(c) *Colours of Slag Powders*

Various American operators make use of the colours of slag powders as an indication of the lime/silica ratios. Tear [13] compares powdered slag samples with standard colour samples. Soler [13] says that he finds a definite change in the colour of the slag powder at a lime/silica ratio of about 2 to 1. Malloy [7] describes the colours of a series of 16 powdered slags. At a lime/silica ratio of approximately 2 to 1 the colour is described as changing from dark tan to very light brown.

(d) *Microscopic Examination*

Twenty years ago Andrew and Hyslop [14] examined basic open-hearth slags petrologically. Much more recently McCaughey,[15] Smith,[16] and Singewald [17] have reported results of petrographic examinations which show the sequence of changes in the constitution of the slag as refining progresses. Since the time required for the preparation of thin sections prohibits the use of this method for slag control, Tenenbaum and Joseph [18] examined basic open-hearth slags in reflected light. Suitable samples for this type of observation may be prepared in less than 5 min. Although the short time required to prepare slag surfaces for examination in reflected light would be very advantageous in control work, this method does not reveal colour contrasts, which are very helpful in the identification of phases in thin sections. From the early acid slags to the very basic finishing slags a rather definite sequence of structural changes takes place. Tenenbaum and Joseph say that, for any particular plant, standard microstructures could be established for slags from heats of various grades of steel.

Remarks

Examination of the foregoing information has led the author to combine the principles of the methods based on a knowledge of the chemical and physical properties described in (a) and (b) above.

For any method of slag control which depends upon the chemical analysis of a slag, the slag must be in a well-fluxed condition when the sample is taken. Although Bacon [1] has found that the slag and metal composition show very little variation during the last 30 min. of the melting period, the author feels that it is far better to take the samples at melting, because such samples are more representative of the slag and metal conditions and also because it is not easy to forecast accurately when the metal will be completely melted. Various methods for the rapid chemical determination of the constituents of basic open-hearth slags have been described and, by using the methods outlined below, the Fe, SiO_2, P_2O_5, and CaO contents may be determined within 30 min. of the sample being taken from the furnace (the CaO content is not required in the calculation of the feed). From the appearance of a slag pancake it is possible to form a reasonably good estimate of the limestone requirements of a charge after melting. That is, during the half hour after melting, before the chemical composition of the slag is known, some progress in the working of the charge may be made in the way of making limestone additions if such additions are required. Also, such a procedure gives a slag which, in the early refining stages, is sufficiently basic and is low in Fe content. These conditions help the removal of sulphur.

Slag-Control Procedure at Park Gate

The steelmaking plant is a hot-metal shop consisting of a 450-ton active mixer and ten basic open-hearth fixed furnaces ranging in capacity from 50 to 95 tons. The average chemical composition of the pig iron is as follows :

C, %	Si, %	S, %	P, %	Mn, %
3·8	0·8	0·06	1·6	1·4

The pig iron is conveyed molten in 25-ton ladles to the mixer. A certain amount of scrap is melted in the mixer and suitable amounts of ore are fed in order that the silicon content of the metal drawn from the mixer may be kept as near as possible to a suitable predetermined figure. In order that the amounts of slag carried in the steelmaking furnaces shall not be too bulky, the silicon content of the mixer metal is made less as the percentage

of hot metal used in the open-hearth furnace increases; for example, when using 45% of hot metal the desired silicon content is 0·3–0·4% and when using 35–40% of hot metal the desired silicon content is 0·4–0·5%. The percentage contents of other elements in the metal withdrawn from the mixer are as follows:

C, %	S, %	P, %	Mn, %
2·8	0·055	1·4	1·0

Owing to the fluctuations in the chemical composition of the iron from the blast-furnaces, and since there are times when heavy demands are made on the mixer, there is difficulty in keeping the analysis of the mixer metal constant. Usually the amount of mixer metal used in the basic open-hearth furnaces is 40–45%.

Suitable amounts of limestone, burnt lime, and ore are put in the furnace with the charge in an endeavour to melt low in phosphorus and with a melter carbon 0·35–0·40% higher than the figure to be obtained at tapping. Limestone together with some burnt lime is used with the charge, because if all limestone is used the time required for melting may be adversely affected.

When the charge is completely melted and the slag is in a well-fluxed condition, samples are taken. A slag pancake is obtained for visual examination and slag and metal samples are taken for analysis.

(1) *Visual Examination of Slag Pancake*

The amount of limestone required after melting is estimated from the appearance of a melting-slag pancake, and any such addition is made immediately. A pancake is produced by allowing a slag sample to solidify in a cast-iron mould, the internal depth of which is $\frac{7}{8}$ in. and the internal diameters $4\frac{1}{2}$ in. at the top and 4 in. at the bottom. A slag which has a bright bottom surface and a top surface free from wrinkles indicates that large limestone additions are required. As the slag becomes more basic the top surface of the pancake develops a wrinkled appearance, the bottom remaining bright. With further limestone additions the wrinkles gradually disappear and the bottom becomes increasingly dull. After the top surface has become smooth the limestone requirements must be estimated from the relative areas of bright and dull spots on the bottom surfaces. Limestone additions are required until the whole of the bottom surface is dull.

(2) *Chemical Analysis of Basic Slag for Iron, Silica, Phosphorus Pentoxide, and Lime*

Sampling

Half the pancake slag which is taken for analysis is pounded in an iron mortar and the major portion put through a 60-mesh sieve. A magnet is run through the sample in order to abstract any metallic shot that may still be present.

Analytical Procedure

Iron—Weigh 1 g. of the crushed sieved slag into a 400-ml. beaker. Dissolve in 50 ml. of hydrochloric acid (sp. gr. 1·16). When dissolved, reduce the coloured solution with stannous chloride, drop by drop from a glass tube, until all the iron is reduced, finally adding three or four drops in excess. Dilute with water, cool, and add mercuric chloride solution to convert excess stannous chloride to stannic chloride. Add a few millilitres of indicator, and titrate with potassium dichromate until a purple colour is obtained. One millilitre of potassium dichromate is equivalent to 1·0% of Fe on the 1-g. sample.

Silica—Weigh 0·5 g. of the crushed sieved slag into a dry 250-ml. squat Pyrex beaker. Add the least possible amount, 15 ml. maximum, of hydrochloric acid (sp. gr. 1·16) and put the whole on to a gas hot-plate. After boiling for a few minutes, add 5 ml. of nitric acid (sp. gr. 1·42), take the assay to dryness, and bake as quickly as possible. After baking, cool slightly, add 20 ml. of hydrochloric acid (sp. gr. 1·16), boil for a few minutes, and then dilute with 15 ml. of hot water. Filter through an ashless paper pad, bobby out the beaker thoroughly with hydrochloric acid (sp. gr. 1·16), and wash once with hot water. Then wash once more with cold hydrochloric acid (sp. gr. 1·16) and finally remove all acids with hot water. Free the pad of water by means of one washing with industrial spirit, and burn off to silica in a flat-bottomed crucible in front of a hot muffle. The weight of the silica precipitate (in grammes) multiplied by 200 gives the percentage of SiO_2 in the sample.

If the precipitate of silica is contaminated with iron or chromium (which condition is easily discernible from the colour of the precipitate), treat with two drops of sulphuric acid (sp. gr. 1·84) and 5 ml. of hydrofluoric acid in a platinum crucible, fuming and baking to expel excess acids. The weight of the residue is subtracted from the weight of the original precipitate, and the resulting weight, when multiplied by 200, gives the true percentage silica content.

Phosphorus Pentoxide—Weigh 0·2 g. of the crushed sieved slag into a 250-ml. conical beaker. Add 10 ml. of nitric acid (sp. gr. 1·20) and bring the liquid up to the boil. The slag should all be in solution by this time. Filter the liquid through a single rapid paper and wash two or three times with a fine jet of hot water. To the filtrate add 15 ml. of hot ammonium nitrate solution followed

by 25 ml. of 10% ammonium molybdate solution, and shake the whole for about 1 min. (The ammonium nitrate solution is made by neutralizing 1740 ml. of nitric acid (sp. gr. 1·20) with 800 ml. of ammonium hydrate (sp. gr. 0·88) and adding 20 g. of ammonium chloride.) Without allowing to stand, filter off through a single rapid paper, washing the precipitate with 2% nitric acid, and then wash acid-free with 2% potassium nitrate solution. Transfer the paper and contents to a 250-ml. conical beaker and titrate with standard caustic soda and nitric acid solutions as used for Handy's phosphorus method. The number of millilitres of caustic soda solution multiplied by 0·229 gives the percentage of P_2O_5 in the sample.

Lime—Weigh 0·5 g. of the crushed sieved slag into a 600-ml. conical beaker. Add 200 ml. of hot water and dissolve the sample by adding 10 ml. of nitric acid (sp. gr. 1·42). Add ammonium hydroxide cautiously until the solution is just neutral. If a precipitate forms, redissolve it in hydrochloric acid (sp. gr. 1·16). Boil, and add crystals of ammonium oxalate until a precipitate of calcium oxalate is obtained. Add a few crystals in excess. Boil until all the crystals are in solution and allow to stand for a few minutes. Filter through a No. 40 Whatman paper 12·5 cm. in dia., and wash well with hot water. Wash the precipitate back into the conical vessel by means of a jet of hot water. Dilute with 100 ml. of water and dissolve the precipitate with 10 ml. of sulphuric acid (sp. gr. 1·84). Bring to the boil and titrate with a standard solution of potassium permanganate, 1 ml. of which is equal to 1% of CaO on the 0·5-g. sample.

(3) *Calculations for Required Additions*

As the analytical results become available in the laboratory, a clerical worker, aided by charts and graphs, quickly calculates the required additions. Bacon [1] has given details of such calculations, but the following example will serve to show the procedure :

Cast T *5749*

	C, %	Si, %	S, %	P, %	Mn, %
Specification ...	0·20–0·25	0·1–0·2	0·05 max.	0·05 max.	0·75–0·85

Chemical compositions of slag and metal samples taken at melting (12.30 A.M.) :

	C, %	S, %	P, %
Metal	0·45	0·049	0·091

	SiO_2, %	CaO, %	P_2O_5, %	Fe, %
Slag	17·2	58·6	9·6	4·9

	Tons	Cwt.
Metallic charged weight	100	5
Limestone (charged)	3	0
Burnt lime (charged)	1	17
∴ Total equivalent limestone (charged)	6	14

Calculation of Limestone Required

The phosphorus which is to be removed from the bath is converted to P_2O_5.

$$\text{“}P_2O_5\text{ correction”} = \frac{(P - 0\cdot 03) \times 2\cdot 3 \times \text{Metallic charged weight}}{\text{Weight of melting slag}} = \frac{0\cdot 061 \times 2\cdot 3 \times 100\cdot 25}{6\cdot 7} = 2\cdot 1\%.$$

$$\therefore \text{Total acids} = SiO_2 + P_2O_5 + \text{“}P_2O_5\text{ correction”} = 17\cdot 2 + 9\cdot 6 + 2\cdot 1 = 28\cdot 9\%.$$

The weight of tapping slag required in order to dilute these " total acids " to 18% is :

$$\frac{\text{Total acids in melting slag} \times \text{Weight of melting slag}}{18} = \frac{28\cdot 9 \times 134}{18} = 215 \text{ cwt.}$$

The amount of limestone required after melting, to give such a tapping slag, is calculated by subtracting from the weight of the tapping slag the total weight of the melting slag and the estimated equivalent limestone picked up from the banks. (The equivalent amount of limestone obtained from the dolomite is estimated from a previous survey of the amount of dolomite used for fettling.)

$$\therefore \text{Limestone required after melting} = 215 - 134 - 36 = 45 \text{ cwt.}$$

At melting, 30 cwt. (2 boxes) of limestone were added after a visual examination of the pancake sample, and therefore a further 15 cwt. (1 box) were added after the calculations had been made.

Calculation of Oxides Required

To remove 0·01% of carbon or phosphorus from a 90-ton bath requires 0·87 cwt. of ore. From the calculated weight of ore an amount equivalent to that supplied by furnace oxidation must be deducted, and for the furnace in question this is equal to 10·5 cwt. of ore for each hour of refining time.

$$\text{C to lose} = 0\cdot 45\% - 0\cdot 10\% = 0\cdot 35\%$$
$$\text{P to lose} = 0\cdot 091\% - 0\cdot 03\% = 0\cdot 061\%$$
$$\therefore (C + P) \text{ to lose} = 0\cdot 411\%.$$

Estimated refining time : 2 hr.

$$\therefore \text{Ore required} = 41\cdot 1 \times 0\cdot 87 - 2 \times 10\cdot 5 = 14\cdot 7 \text{ cwt.}$$

To raise the slag Fe by 1% for each 1 ton of slag, 0·24 cwt. of scale is required. The amount of scale required to raise the slag Fe to 9% is calculated, because, if the slag Fe is 9% at the time when the final metal sample is taken, it will be 11–12% by the time the furnace is tapped.

$$\therefore \text{Scale required} = 0\cdot 24 \times \text{Weight (tons) of tapping slag} \times \text{Desired increment of slag Fe} = 0\cdot 24 \times 10\cdot 75 \times (9 - 4\cdot 9) = 10\cdot 6 \text{ cwt.}$$

The above additions were made, and the charge was tapped at 2·45 A.M. (refining time, 2 hr. 15 min.). The chemical compositions of the final bath sample, test ingot, and tapping slag were as follows :

	C, %	Si, %	S, %	P, %	Mn, %
Final bath sample ...	0·10	...	0·031	0·030	0·20
Test ingot	0·23	0·15	0·032	0·026	0·85

	SiO_2, %	CaO, %	P_2O_5, %	Fe, %
Tapping slag...	11·0	51·6	7·73	11·9

(4) *Application of Slag Control in the Manufacture of Various Types of Steel*

The above principles are applied to the manufacture of all the steel made at Park Gate. There are, of course, differences in detail according to the type of steel being made. The details shown in the above example are closely followed in making the lower-carbon steels, that is, the desired slag Fe used in the calculation is 9% and scale is used to obtain the increase in slag Fe, whilst ore is used to remove the carbon and phosphorus from the bath. For all steels with over 0·25% of carbon the carbon content of the bath at tapping is " caught " at one or two " points " (hundredths of one per cent.) below the percentage desired in the ingots. The amount of ore required to bring the carbon down to the top of the specification is calculated, but in the case of " catchers " scale is used in lieu of ore. Scale is used to raise the slag Fe to the required value, the desired slag Fe used in such a calculation being 11%. The reason for using a " desired slag Fe " of 9% in the case of low-carbon steels and 11% for the " catchers " is that for the former the slag Fe increases from 9% to 11 or 12% during the time in which the final metal sample is being analysed, whereas for the latter carbon is being removed during this period and consequently there is less tendency for the slag Fe to increase.

In the manufacture of high-sulphur free-cutting steels the tapping-slag Fe is higher, and the total acids in the tapping slag may be 21%.

Rimming steels are made by this method of slag control, using a suitable " desired slag Fe."

Investigation of Slag Analyses of Casts made by the Application of Slag Control

In order to obtain information relating to the chemical compositions and basicities of slags formed when making steel by the application of slag control, a statistical survey of 250 casts has been made. Although all the steel produced at Park Gate is made according to the slag-control method described earlier, in order to make the investigation of maximum value the casts selected for examination fulfilled the following conditions :

(*a*) The casts were all of plain carbon steels, the manganese contents of which did not exceed 0·90%.

(*b*) All the casts rolled well and were free from serious surface defects in the billet stage.

(*c*) Analysis of the melting slags and subsequent calculations indicated that the slags had the correct lime content or that limestone was required after melting. That is, no cast of which the melting slag contained lime in excess

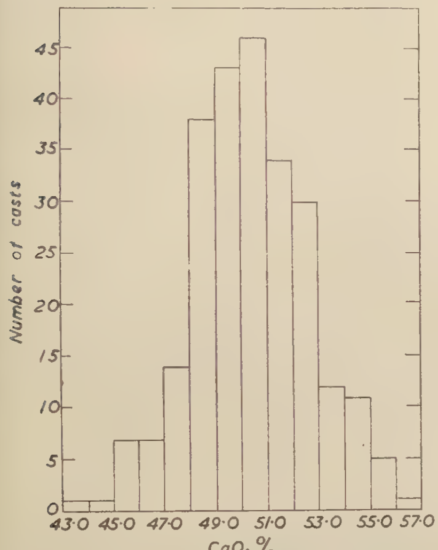

Fig. 1—CaO contents of tapping slags

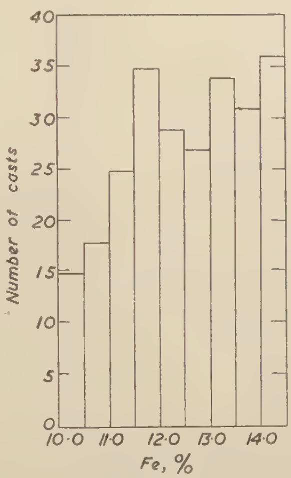

Fig. 2—Fe contents of tapping slags

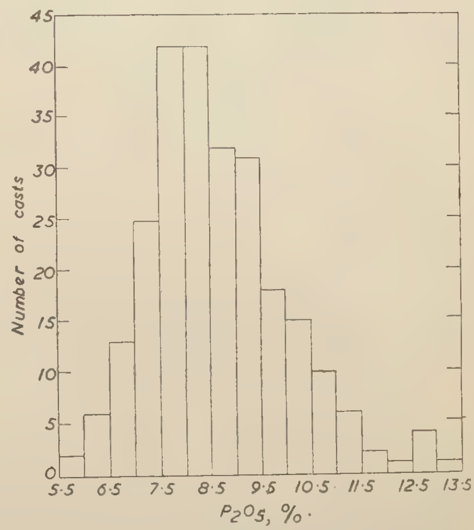

Fig. 3—P_2O_5 contents of tapping slags

of refining requirements was included in the survey.

(*d*) The results did not include any cast which had a tapping-slag Fe greater than 14·5%.

Figures 1 to 4 are histograms showing the CaO, Fe, P_2O_5, and SiO_2 contents of the tapping slags. Most of the tapping slags contained between 10·0 and 11·5% of SiO_2, and between 7·5 and 9·5% of P_2O_5. The CaO content shows a peak at 50–51%, and only a few of the tapping slags have a CaO content of less than 48% or more than 53%. Figure 2 shows that the Fe content of the tapping slags is usually between 11·5 and 14·5%, and the distribution over this range is approximately constant. The slag-control calculation aims at the (SiO_2 + P_2O_5) content of the tapping slag being 18%. Figure 5 shows that very few of the tapping slags finish with these total acids less than 17·5% or greater than 20·5%.

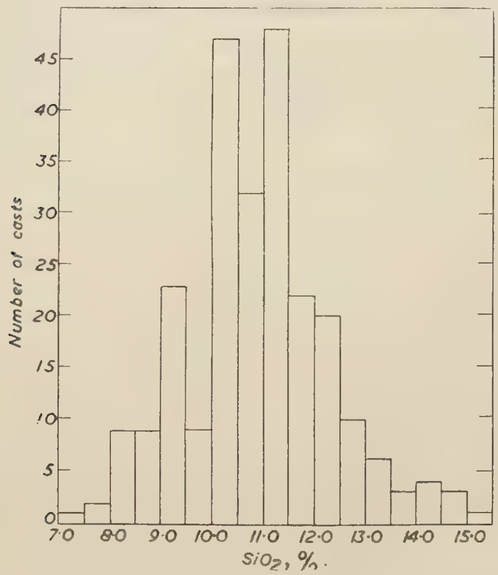

Fig. 4—SiO_2 contents of tapping slags

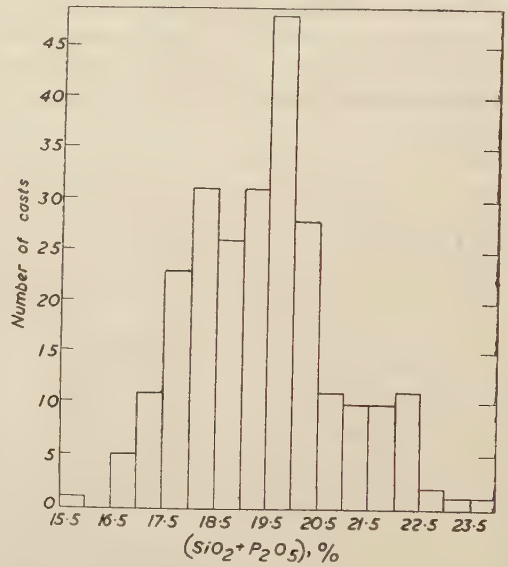

Fig. 5—Total acid contents of tapping slags

Measurement of Basicities of Basic Open-Hearth Slags

There are various ways, using chemical compositions, by which the basicity of slags may be expressed. For a long time it has been customary in America to express basicity as the ratio of lime to silica, and this is frequently designated as the "*V*" value. For British practice, where the slags frequently contain high percentages of P_2O_5, the basicity value must take into account such P_2O_5 content. If we assume the formation of tricalcium phosphate then a corrected lime/silica ratio may be expressed as (CaO — 1·18 P_2O_5)/SiO_2, and reference has been made to such a ratio by Reagan [19] and others.[20] Fetters and Chipman,[21] in using a corrected ratio of CaO/(SiO_2 + 0·634 P_2O_5), say that "this method of correcting for phosphorus assumes that one mol of P_2O_5 can combine with half again as much lime as one mol of SiO_2; it seems more reasonable to add this to the silica than to subtract it from the lime." Another way of expressing basicity is CaO/(SiO_2+ P_2O_5). This expression has been used by Bacon,[1] Rait and Goldschmidt,[22] and others.[20] Since so many compounds are formed in basic open-hearth slags it is difficult to define what is meant by the basicity of a slag, and, therefore, different expressions have found favour in attempts to indicate relative basicities. Both the melting and tapping slags of the 250 casts under review were analysed, and in each case the basicities were calculated using each of the three expressions (CaO — 1·18 P_2O_5)/SiO_2, CaO/(SiO_2 + 0·634 P_2O_5), and CaO/(SiO_2 + P_2O_5). An examination of such basicities of the tapping slags has been made in order to find the optimum values of tapping-slag basicities and the range of such values. Various opinions have been expressed with reference to the optimum value of the lime/silica ratio. Most of these opinions relate to American practice, in which the P_2O_5 contents of the tapping slags are low, and therefore one would expect the desired lime/silica ratio to be standardized. Washburn and Philbrook [8] say that the charge should be slightly deficient in lime, additions of burnt lime being made as necessary, to maintain a basicity of 2·2–2·5 "*V*" value during the refining period. Then the slag should be thickened by additions of burnt lime just before charging furnace

deoxidizers, in order to decrease the rate of transfer of FeO to the bath. Mauer [11] says that the lime/silica ratio at melt should be 1·8–2·0, with some undissolved lime floating in the slag, and during the later stages of the refining period the lime/silica ratio should be 2·3–2·5. In dealing with the use of slag control for making recarburized rail steel, Miller and Washburn [23] say the aim is to ensure the attainment of a lime/silica ratio of at least 2·8 to 1 at 0·30% of carbon, and one of 3·1 to 1 at 0·15% of carbon when the bath is ready for its metal recarburizer. Knight [4] says that for the production of low- and medium-carbon semi-killed steels at Kembla the finishing slags are run to a lime/silica ratio of 2·0–2·2. In the production of alloy and forging steels the lime/silica ratios vary between 2·5 and 2·7. The P_2O_5 carried in the slag averages only 1%.

Iwanow [24] concluded that it is necessary to work with a low-viscosity and not too highly basic slag having a lime/silica ratio of from 1·8 to 2·0 in order to produce a high-quality open-hearth steel.

At the 1939 American Open-Hearth Conference[13] several operators gave their usual lime/silica ratios, as follows:

G. Soler: "Ratio 2·8 to 1; rather that is about 2·6 to 1, when corrected for phosphorus pentoxide."

W. P. Albaugh: "On some grades we use a ratio of 2½ to 1; on other grades, 4 to 1."

T. S. Washburn: "On the strip grades the ratio is about 3·1 to 1; on forging grades, about 2·6 to 1."

H. L. Tear: "On our killed steels, depending on how we kill them, we never go below 2·6."

Figure 6 is a histogram showing the values of basicity, as measured by $(CaO - 1\cdot18\ P_2O_5)/SiO_2$, of the tapping slags. The basicities cover a very wide range from 2·5 to 5·5, and few casts have tapping-slag basicities less than 3·2 or greater than 4·3.

Figure 7 shows the distribution of basicities of the tapping slags as measured by $CaO/(SiO_2 + 0\cdot634\ P_2O_5)$. The total range of values is from 2·3 to 4·0, with the majority of basicities lying between 2·8 and 3·3. Figure 7 shows a much better distribution than that shown in Fig. 6.

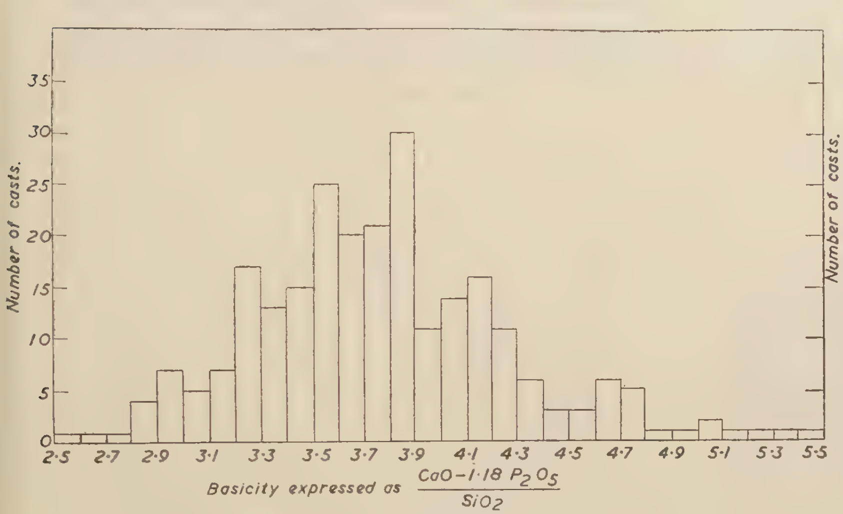

FIG. 6—Basicities of tapping slags as measured by $(CaO - 1\cdot18\ P_2O_5)/SiO_2$

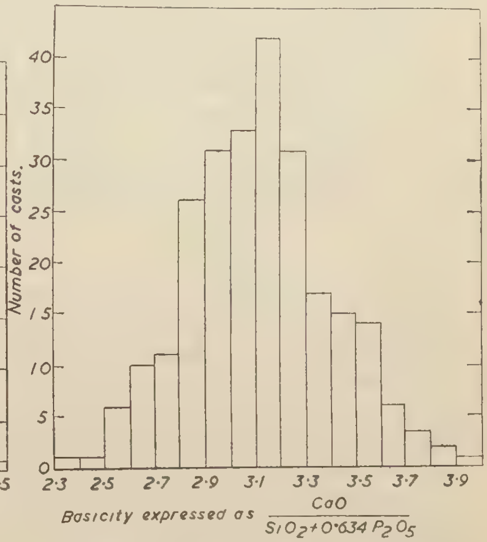

FIG. 7—Basicities of tapping slags as measured by $CaO/(SiO_2 + 0\cdot634\ P_2O_5)$

Figure 8 shows the distribution of basicities of the tapping slags as measured by $CaO/(SiO_2 + P_2O_5)$. The values range from 2·0 to 3·2, with a preponderance of values between 2·5 and 2·8. From Figs. 6, 7, and 8 it is deduced that the best of the three ways of measuring basicity is by $CaO/(SiO_2 + P_2O_5)$, and that most of the tapping-slag basicities lie between 2·5 and 2·8. The peak on the CaO histogram (Fig. 1) is 50·5% and the peak on the $(SiO_2 + P_2O_5)$ histogram (Fig. 5) is 19·75%. Division of these values gives a basicity of 2·56. The slag-control calculations aim at $(SiO_2 + P_2O_5)$ in the tapping slag being 18%, and if this figure is divided into the optimum CaO value of 50·5% a basicity of 2·8 is obtained.

Correlation of Slag Basicities as Determined by Different Formulæ

If there is any true relationship between the values of the basicity of a slag obtained from two different formulæ, then a graph connecting such

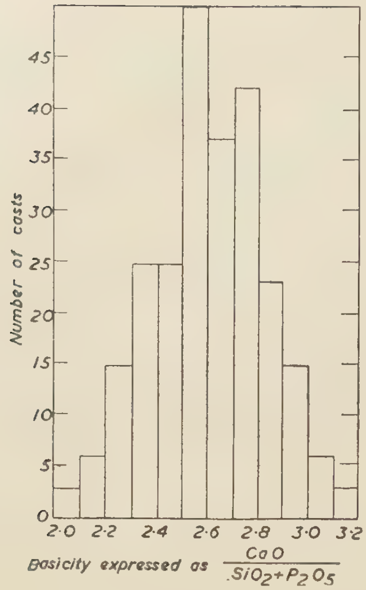

FIG. 8—Basicities of tapping slags as measured by $CaO/(SiO_2 + P_2O_5)$

values for a number of slags should be a smooth curve (or a straight line). Therefore, the basicities of the *melting* slags of the 250 casts under review have been calculated using the three different formulæ, and Figs. 9, 10, and 11 show the results of plotting the three values of basicities against each other. The basicities of *melting* slags have been used so that a much wider range of basicities could be examined than would have been the case had the basicities of the tapping slags been plotted.

Figure 9 shows that for low basicities there is a straight-line relationship between the values of basicities measured by $(CaO - 1 \cdot 18\ P_2O_5)/SiO_2$ and $CaO/(SiO_2 + 0 \cdot 634\ P_2O_5)$. Scatter of the points becomes pronounced at the higher basicity values. Figure 10 shows much scatter from a straight-line relationship of basicity values measured by $CaO/(SiO_2 + 0 \cdot 634\ P_2O_5)$ and $CaO/(SiO_2 + P_2O_5)$. Figure 11 shows very little relationship between the basicity values measured by $(CaO - 1 \cdot 18\ P_2O_5)/SiO_2$ and $CaO/(SiO_2 + P_2O_5)$. Figures 9, 10, and 11 show that at the higher basicity values of the melting slags there is very little correlation between the basicity values as measured by the three different formulæ.

Relationship between Basicity of Melting Slag and Limestone Addition Required after Melting

Of the 250 casts under review, 66 were made in *T* furnace (95 tons capacity). The amounts of limestone required after melting, as worked out by the slag-control method described earlier, are plotted against the melting-slag basicities in Figs. 12, 13, and 14. The amounts of limestone include the "dolomite allowances," which, of course, must be deducted from the figures given. The graphs give approximately straight-line relationships. In the slag-control method described earlier an estimate of limestone requirements after melting is made from a visual examination of the melting-slag pancake. Such an estimation is made in terms of boxes (15 cwt.) of limestone, and, whilst experience enables one to make reasonably accurate estimations, it has not been found possible to assign accurate basicity values to slag pancakes as a result of visual examination. Hence it has not been possible to supplement the existing slag-control procedure by graphs such as Fig. 12. Any estimation made from a visual examination of a melting-slag

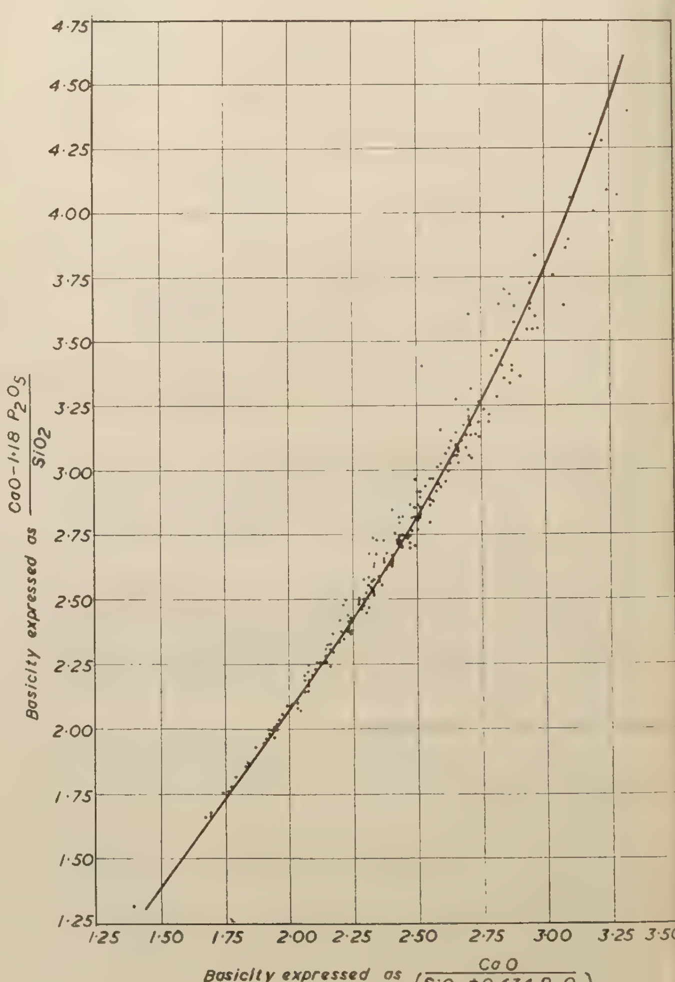

FIG. 9—Relationship between $(CaO - 1 \cdot 18\ P_2O_5)/SiO$, and $CaO/(SiO_2 + 0 \cdot 634\ P_2O_5)$ in melting slags

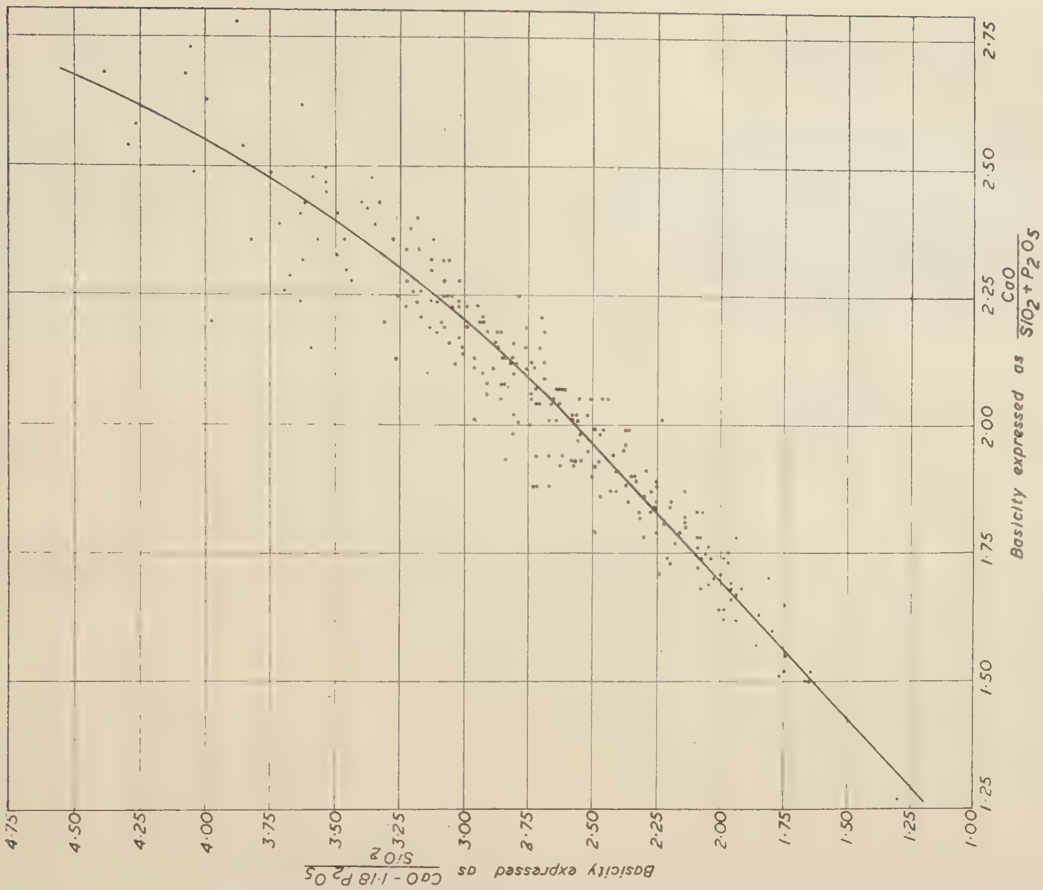

FIG. 11—Relationship between $(CaO - 1 \cdot 18\ P_2O_5)/SiO_2$ and $CaO/(SiO_2 + P_2O_5)$ in melting slags

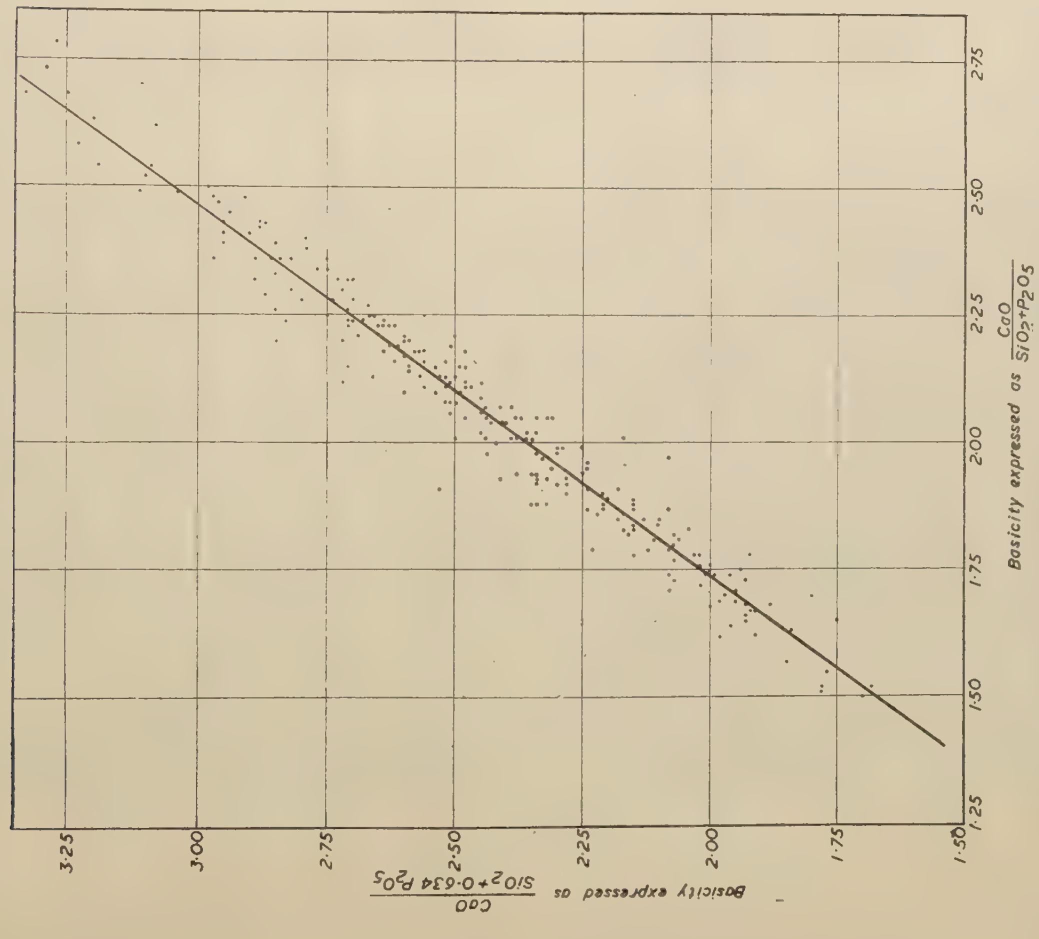

FIG. 10—Relationship between $CaO/(SiO_2 + 0 \cdot 634\ P_2O_5)$ and $CaO/(SiO_2 + P_2O_5)$ in melting slags

pancake is, of course, taken into account 30 min. after melting, when the calculated feed requirements are known.

Summary and Conclusions

(1) A method of slag control which is based on a combination of existing practices is described. The slag and metal samples are taken when the bath is clear-melted and a well-fluxed slag has been formed.

(2) A well-staffed laboratory (including a clerical worker) enables the feed additions to be calculated within 30 min. of the slag and metal samples being taken from the furnace. Frequently, limestone additions are made during that 30 min.

(3) This slag-control method is applied to the manufacture of all the steel made at Park Gate, and beneficial results are being obtained both in the quantity and quality of the steel produced. The average refining time has been reduced by 37%. Much of this improvement is a direct result of the application of slag control during refining, but some of the improvement is due to the fact that more attention is paid to the amounts of limestone and ore put in with the charge in order to obtain reasonable chemical compositions of metal and slag at melting. Since all the additions are made during the early part of the refining period, a good " boil " is subsequently obtained, and this is essential for the production of good-quality steel.

(4) A statistical survey of analyses of the melting and tapping slags of 250 casts has been made. Histograms give the optimum values of tapping-slag basicities as determined by various expressions. Graphs are given showing that there are no accurate linear relationships between slag basicities as determined by the three expressions $(CaO - 1\cdot18\ P_2O_5)/SiO_2$, $CaO/(SiO_2 + 0\cdot634\ P_2O_5)$, and $CaO/(SiO_2 + P_2O_5)$. The more basic the slag the greater the divergencies from the linear relationships.

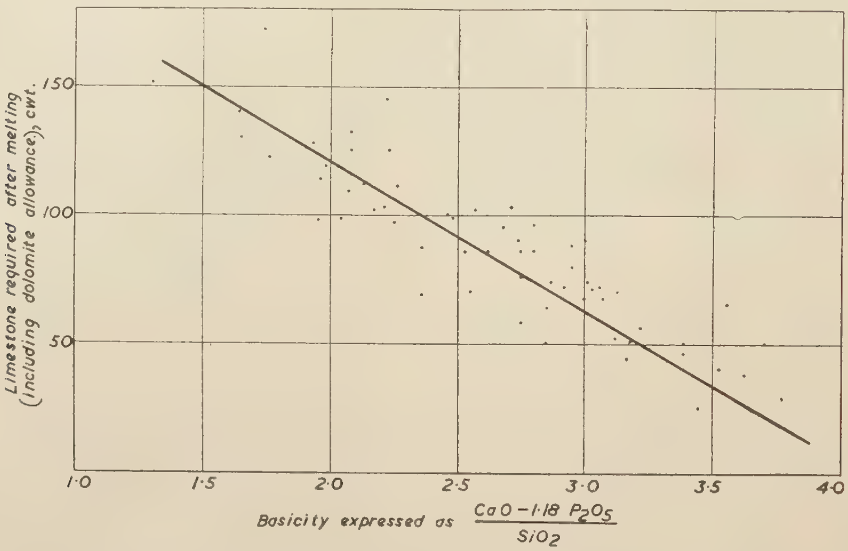

Fig. 12—Relationship between $(CaO-1\cdot18\ P_2O_5)/SiO_2$ and limestone required after melting (*T* furnace)

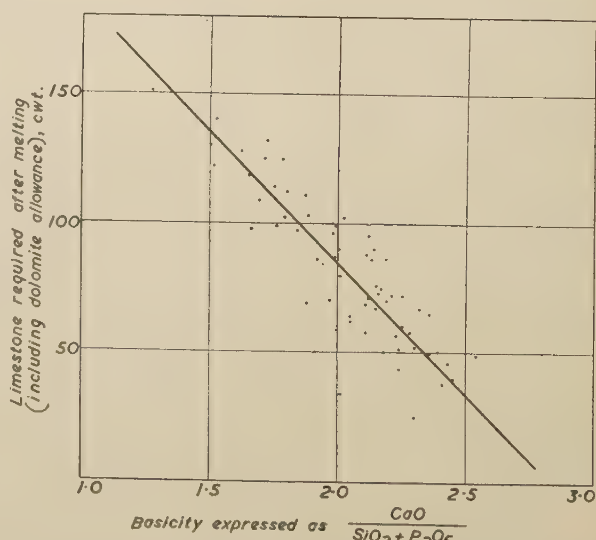

Fig. 13—Relationship between $CaO/(SiO_2 + P_2O_5)$ and limestone required after melting (*T* furnace)

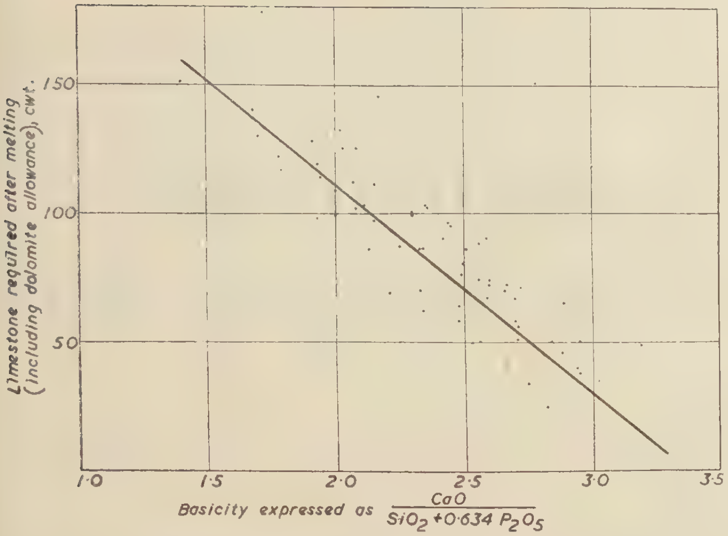

FIG. 14—Relationship between $CaO/(SiO_2 + 0{\cdot}634\ P_2O_5)$ and limestone required after melting (*T* furnace)

ACKNOWLEDGMENTS

The author wishes to express his gratitude to the Directors of The Park Gate Iron and Steel Co., Ltd., for permission to publish the information contained in this paper, and in particular to Mr. H. Williams, Director and General Manager, for his interest in the work. The author is also indebted to those, both staff and workpeople, whose efforts have made possible the successful application of this method of slag control.

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The Microstructure of Commercial Acid-Resisting Silicon-Iron Alloys*

By *J. E. Hurst, D.Met.,*† *and R. V. Riley, B.Sc., Ph.D.*†

SYNOPSIS

Difficulties in the preparation of metallographic specimens and in the interpretation of the microstructures of acid-resisting silicon iron have been largely overcome. The disposition of the phases in commercial irons containing between 14% and 18% of silicon has been found to agree on the whole with what would be expected from a study of the most recent iron–silicon binary diagram. The influence on the microstructure of variations in silicon content, additions of certain alloying elements, and of adventitious impurities has been studied and photomicrographs are presented.

INTRODUCTION

COMMERCIAL acid-resisting silicon-iron alloys with over 14% of silicon are used widely for heavy duty corrosion resistance in the chemical industries. The alloys contain in addition to silicon, other elements the chief of which are carbon, manganese, sulphur, and phosphorus. Certain types of silicon iron designed for special applications may contain also the elements copper, nickel, and molybdenum. Titanium, aluminium, and sometimes nickel, are very often present as residual elements arising from the raw materials used in making the iron alloy.

Very little information has been reported on the metallographic features of these alloys. This paper is concerned with this subject and, in particular, with the influence on the microstructure of the elements manganese, phosphorus, copper, and molybdenum.

To secure adequate resistance to corrosion against the mineral acids the iron should contain approximately 14·5–15% of silicon. The limit of the alpha phase under equilibrium conditions in the binary iron–silicon system at room temperature is about 15·2% of silicon, and although the effect of additional elements such as carbon, phosphorus, sulphur, and manganese is not known precisely, it is sufficient to observe that the necessary minimum silicon content of the commercial acid-resisting alloys is quite close to the upper limit of this alpha phase region. Examples of commercial alloys which contain upwards of 15% of silicon are by no means uncommon; for example, an alloy containing 16–17% of silicon is deliberately produced for resistance to hot hydrochloric acid solutions. A knowledge of the phases existing at or about the boundary composition of 15·2% of silicon is required for a complete understanding of the metallurgy of acid-resisting silicon irons.

According to the latest binary equilibrium diagram[1] for the iron–silicon system (Fig. 1), alloys containing over 15·2% of silicon are duplex and comprise when in thermal equilibrium at room temperature, the alpha and epsilon phases. The epsilon phase is considered to be an intermetallic compound having the composition FeSi. At temperatures in excess of 800° C., and with silicon percentages over 18% the system is further complicated by a modified form of alpha phase and another silicon-containing phase described as the eta phase. The eta phase, which has a stable existence in the temperature range 800°–1000°C.

* Received 12th July, 1946.

† The Staveley/Bradley Foster Research Laboratories, Darlaston.

has been identified frequently both in pure binary alloys and in a range of commercial alloys containing over 15·2% of silicon, prepared in a normal manner. Lipson and Weill[2] have put forward an explanation of why the eta phase tends to appear before the epsilon phase, even outside the limits

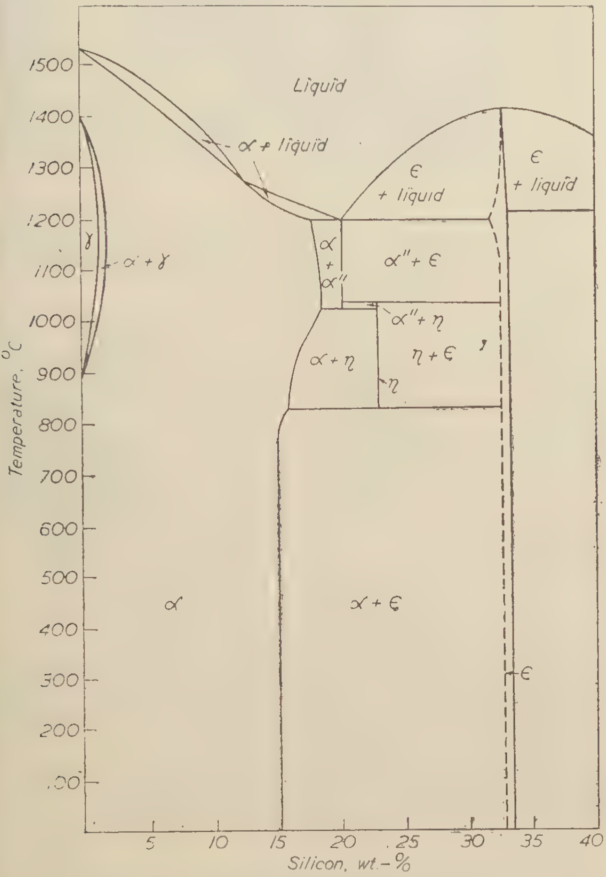

FIG. 1—Binary iron–silicon thermal equilibrium diagram according to Farquhar, Lipson, and Weill

of its existence in the equilibrium diagram. It is, however, necessary to add that in the light of our further experience the epsilon phase is present more often than formerly thought in commercial alloys.

Specimen Preparation

The preparation of silicon-iron-alloy specimens for metallographic examination is difficult ; the hardness of the iron, its proneness to interdendritic porosity, and its rather low mechanical strength all make the specimen liable to scratching during polishing. The etching of the polished surface to reveal the underlying structure is likewise not easy. Owing to the acid-resistant qualities of the metal a very strong etchant has to be employed and difficulty may then be experienced in obtaining a true structure of the alloy without interference from pseudo film-like surface markings. One manifestation of such an incorrect structure may sometimes be seen when using mixtures containing strong hydrofluoric acid solutions ; this has been described as the ‘ barley shell ’ structure.[3]

The most successful method of specimen preparation for acid-resisting high-silicon alloys is as follows :

(*a*) *Grinding and Polishing*—The specimen is ground flat on an emery wheel and is then smoothed in an orthodox manner on graded emery paper. The finely ground sample is polished on a Selvyt-covered wheel rotating at 500 r.p.m. The polishing cloth is treated with a fairly thick suspension of diamantine powder in water.

(*b*) *Etching*—The polished specimen is etched by immersion for 5 to 20 sec. in a cold solution consisting of 1 part of hydrofluoric acid (A.R.), 1 part of nitric acid, and 6 parts of water. A dip in acetone followed by quick drying in a current of warm air completes the process of specimen preparation.

If it is desired to produce a very high quality microsample suitable for photography, it may be necessary to repeat the polishing and etching several times to eradicate all scratches.

The etchant described above has been found to be a fairly satisfactory all-purpose reagent. This aqueous nitro-hydrofluoric acid reagent is, however, not ideal for a differentiation between the eta and epsilon phases, but despite extensive experimental work it has not been possible to find a better alternative etchant for this purpose. All the photomicrographs shown in this paper, except those taken by polarized light, were prepared from specimens treated in the manner just described. Specimens for examination by polarized light were not etched. Although the nitric–hydrofluoric acid reagent is considered the best all-purpose etchant a fairly satisfactory etch may be obtained by boiling the sample in aqueous sodium picrate solution, but this takes longer and may sometimes adversely affect the specimen mounting material.

The Microstructure of the Main Phases in Silicon-Iron Alloys

(1) *The Alpha Phase*

The alpha phase is the main silicon-containing phase found in silicon-iron alloys of less than 15·2% of silicon. It is a light-etching phase as revealed by the normal etchant (1 part of HF : 1

part of HNO_3 : 6 parts of H_2O), and is generally referred to as silico-ferrite. In commercial acid-resisting silicon iron containing carbon, this is usually dispersed in the alpha-phase matrix as fine flake graphite as shown in Fig. 2. The graphite may be present also in the form of coarse flakes, and other forms are recognized, particularly an acicular or rod-like form and also a type showing a network of reticular spots. The average composition of the specimen illustrated in Fig. 2 is given at *A* in Table I.

(2) *The Eta Phase*

The eta phase is frequently encountered in the structures of commercial iron–silicon alloys, whenever the silicon content exceeds 15·2%, and has been identified by X-ray examination. Microscopic examination of specimens etched in the normal strength nitro-hydrofluoric acid reagent reveals the eta constituent to be coloured darker than the alpha phase. Illumination with white light gives the phase a very faint light-grey-brown colour. The coloration appears to be due to a staining effect rather than to a roughening caused by acid attack. A typical example of the eta phase in a commercial silicon iron having the composition *B* given in Table I, is shown in Fig. 3.

TABLE I—*Typical Commercial Acid-Resisting Silicon Irons*

Sample	Silicon, %	Carbon, %	Manganese, %	Phosphorus, %
A	15·0	0·50	0·64	0·034
B	15·6	0·53	0·53	0·087
C	15·5	0·42	0·45	0·062

(3) *The Epsilon Phase*

From our experience so far it would appear that the epsilon phase does not occur in commercial alloys with the same frequency as does the eta phase. A silicon content over 15·2%, which is responsible for producing the eta phase, may, however, also result in epsilon. The appearance of this latter phase under micro-examination is very similar to eta, and in fact the two are not easily differentiated. The illustration Fig. 4 shows the appearance of the epsilon phase ; in this specimen of composition *C* in Table I, the existence of epsilon has been confirmed by X-ray evidence. In the identification of the eta and epsilon phases none of the etching methods tried so far have yielded satisfactory results. Both the alpha and epsilon phases are body-centred cubic, whilst the eta phase is hexagonal[4] and thus anisotropic and exhibits pleochroism when viewed under crossed nicols.[5] This property revealed by a rotating beam of polarized light, affords a means of distinguishing between eta and the alpha or epsilon phase. Figures 5 and 6 show a polished unetched specimen of a binary alloy containing 27% of silicon almost wholly composed of eta constituent, photographed under the polarizing microscope with the analyser placed $+5°$ and $-5°$ from the crossed position, respectively. The anisotropy of this phase is evident.

The three main silicon-bearing phases in commercial silicon irons have other characteristics which help in their identification under the microscope. A summary of their main points of difference is given in Table II.

In the binary iron–silicon thermal-equilibrium diagram shown in Fig. 1 is a modified alpha phase designated as α'' which has apparently a limited stability at temperatures in excess of 1000° C. Samples of commercial silicon-iron alloys, containing 19–25% of silicon have been quenched in water and chill-cast in thin sections in copper moulds, with the object of retaining this phase for identification under the microscope, but so far it has not been possible separately to identify α'' in this manner. This modification of the alpha phase which has been identified by X-rays in the binary alloy must either not have been retained in the treatment accorded to specimens of commercial alloys in this investigation, or its appearance under the microscope may not have differed sufficiently from that of the normal alpha phase to permit identification in this manner.

THE INFLUENCE OF PHOSPHORUS ON THE MICROSTRUCTURE

For the study of the effect of phosphorus on commercial acid-resisting 14–16% silicon iron, a series of alloys were prepared having the compositions given in Table III. All the specimens were taken from alloys which had been melted at least twice to produce sound gas-free metal. The phosphorus additions in amounts increasing up to 4·18% were introduced by means of ferro-phosphorus. In both the rocking arc furnace and the high-frequency induction furnace the metal was superheated to a temperature between 1450° and 1500° C. (measured by optical pyrometer) before tapping. Microspecimens were taken from sand-cast test bars of ½-in. dia.

It was not possible to reveal effects on the microstructure of the 14–16% silicon alloys of phosphorus in amounts less than 0·1%. The first effect of phosphorus in these experiments was

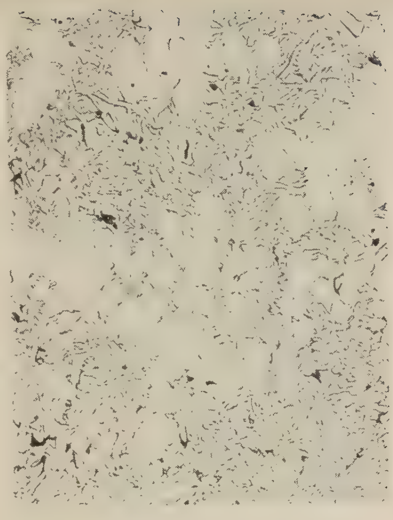

Fig. 2—Fine graphite in a single alpha-phase commercial silicon iron of composition *A*, Table I × 120

Fig. 3—Eta and alpha phases, together with fine graphite in a commercial silicon iron of composition *B*, Table I × 600

Fig. 4—The epsilon phase associated with holes at the boundaries of the alpha grains in an alloy of composition *C*, Table I × 600

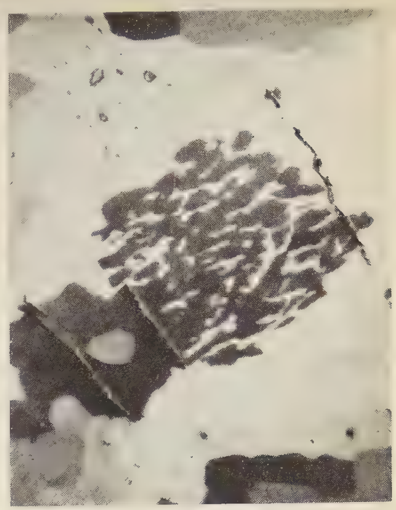

Fig. 5—The appearance of the eta phase under polarized light +5° from the crossed position (*cf.* Fig. 6) × 1200

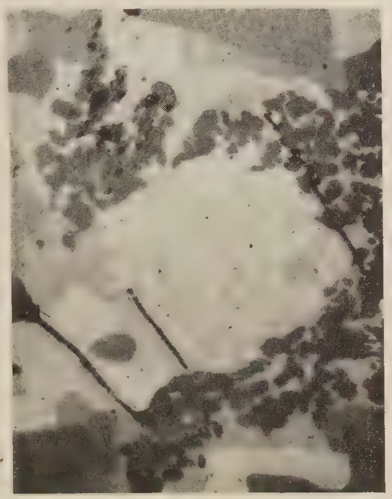

Fig. 6—The appearance of the eta phase under polarized light −5° from the crossed position × 1200

Fig. 7—A typical example of the phosphide carbide complex at the boundaries of the alpha phase in silicon iron of composition *E*, Table III. Note the dispersed phase and the fine spot-like boundary graphite × 600

Fig. 8—Another example of phosphide–carbide complex and the dispersed phase in a silicon iron alloy containing 2·42% of phosphorus; sample *I*, Table III × 600

Fig. 9—Same specimen as in Fig. 8. The breakdown of the carbide–phosphide complex on annealing at 850° C × 600

The microsections appearing in Figs. 2–4 and 7–9 were etched in the nitro-hydrofluoric acid reagent (Figs. 2–9 were reduced to four-fifths linear in reproduction)

[*Hurst and Riley*
[*To face p. 174*

FIG. 10—The eutectiform appearance of the phosphide phase in a hyper-eutectic high phosphorus commercial silicon iron of composition as in specimens *G*, *K*, and *L*, Table III × 600

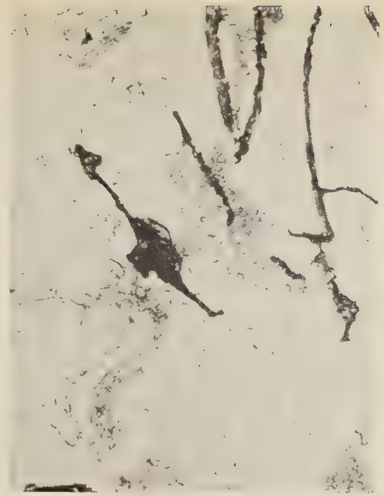

FIG. 11—The eta phase in association with eutectiform phosphide phase in an alloy of composition *L*, Table III × 600

FIG. 12—Specimen *G*, Table III, heated to 1050° C., soaked for 4 hr. and quenched in water. Note the absence of the dispersed phase in the alpha grains × 600

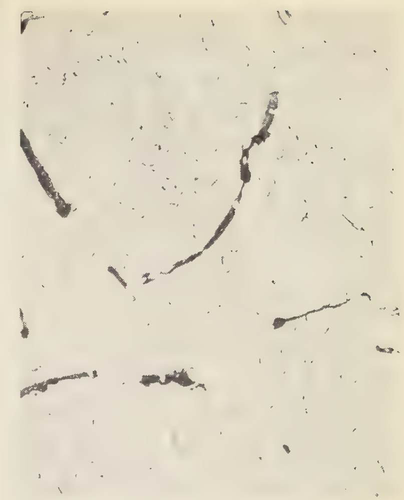

FIG. 13—Same specimen as in Fig. 12, reheated to 1050° C., soaked for 4 hr. and slowly cooled. The dispersed phase again visible as in cast alloy × 600

FIG. 14—Sample *L*. Table III, (*see* Fig. 11) after 3 weeks' annealing at 800° C. Note the agglomeration of the dispersed phase × 600

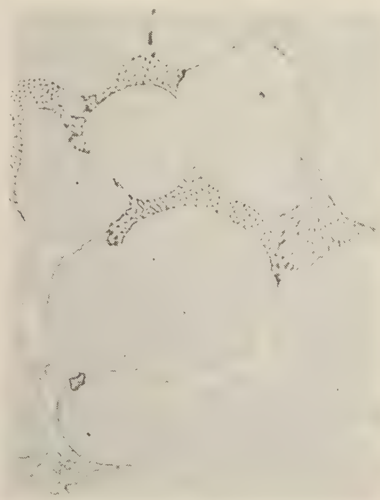

FIG. 15—The partial stabilization of the carbide phase by 7·3% of manganese as in specimen *E*, Table IV × 600

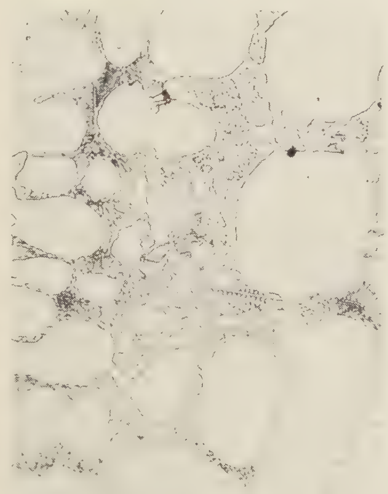

FIG. 16—Specimen *A*, Table V, of commercial silicon iron with 3·5% of molybdenum, showing complete stabilization of the complex carbide × 600

FIG. 17—Same specimen as in Fig. 16, at higher magnification × 1200

The microsections appearing in Figs. 10–17 were etched in the nitro-hydrofluoric acid reagent
(Figs. 10–17 were reduced to four-fifths linear in reproduction)

found with sample *B*, Table III, containing 0·22% of phosphorus. A considerable amount of a dispersed phase and a small amount of light-etching massive phase appeared which was similar to and almost indistinguishable from the carbide phase described by the authors in a previous paper.[6] Figure 7 shows a typical example of this phase. On annealing at 850° C. for 4 hr., the boundary phase suffered partial decomposition. The view is held that in the "as-cast" condition under the conditions of cooling which prevailed in these experiments, a considerable proportion of carbon may be retained in the form of a carbide in association with the phosphide. The carbide–phosphide complex existing in the specimen shown in Fig. 8, broke down on annealing at 850° C. to give a granular type of boundary graphite and a large amount of residual phosphide, Fig. 9.

TABLE II—*Identification of Phases in Commercial Silicon Iron*

Condition	Property	α Phase	η Phase	ε Phase
Polished but unetched	Crystalline nature as indicated by plane-polarized light	Isotropic	Anisotropic	Isotropic
Polished and etched in HF–HNO_3 reagent	Shape of phase and usual mode of occurrence	Usually the matrix in 14% to 18% silicon irons	A boundary phase or a flowery dendritic phase, often cracked and associated with holes formed during specimen preparation	Massive triangular-shaped phase. Usually cracked or associated with holes formed during specimen preparation
Polished and etched in HF–HNO_3 reagent	Colour and surface characteristics	Smooth light-coloured metallic phase	Often rough and a tendency to be scratched. Light-grey-brown colour	Smooth 'slag-like,' light-grey colour

TABLE III—*Composition of Specimens of 14–16%-Silicon Iron containing Phosphorus*

Sample	Method of Melting	Phosphorus, %	Silicon, %	Carbon, %	Manganese, %
A	Arc furnace	0·05	15·2	0·50	0·53
B	High-frequency furnace	0·22	15·3	0·53	0·65
C	High-frequency furnace	0·36	15·3	0·40	0·63
D	Arc furnace	0·50	14·8	0·27	0·56
E	High-frequency furnace	0·60	14·3	0·46	0·62
F	High-frequency furnace	0·82	14·7	0·42	0·62
G	Arc furnace	0·83	15·2	0·77	0·56
H	High-frequency furnace	1·40	14·3	0·40	0·55
I	High-frequency furnace	2·42	14·3	0·37	0·54
J	High-frequency furnace	4·18	14·7	0·46	0·54
K	High-frequency furnace	1·08	14·4	0·90	0·55
L	High-frequency furnace	0·56	15·6	0·69	0·47

the phase was a carbide–phosphide complex; the carbide decomposed during annealing, leaving the residual phosphide phase.

An increase in phosphorus was accompanied by an increased amount of the carbide–phosphide phase in the microstructure. Figure 8 shows the microstructure of sample *I*, Table III, containing 2·42% of phosphorus, and in this specimen the graphite flakes have been almost entirely replaced by the carbide–phosphide complex. The reduced amount of graphite and the increased quantity of the carbide–phosphide complex, indicates that

In high phosphorus silicon-iron alloys which contain sufficient carbon to make them hypereutectiferous, as in specimens *G*, *K*, and *L*,

Table III, having 0·77%, 0·90%, and 0·69% of carbon, respectively, the phosphide was more eutectiform in appearance and was non-oriented in respect to the primary graphite. This type of structure, shown in Fig. 10, is typical of that frequently observed in commercial varieties of cupola-melted acid-resisting phosphoric silicon iron. When the silicon content of a phosphoric hypereutectic silicon iron was raised above 15·2% the phosphide phase remained eutectiform and was closely associated with the eta or epsilon phases. The eta phase is clearly shown in Fig. 11, adjoining the phosphide phase in a specimen of composition *L*, Table III.

The microstructure of most of the 'as-cast' phosphorus-containing silicon irons showed a large amount of a dispersed phase in the alpha silico-ferrite grains. This dispersed phase, which may be observed in Figs. 8–12, inclusive, was studied in order to ascertain its nature. The alpha phase always contained the largest amount of the dispersed phase, but it was not apparently limited to this phase ; Figs. 8 and 10 show that it might also occur, although in much smaller amounts, in the carbide–phosphide complex. The dispersed phase may also be seen in the eta phase shown in Fig. 11. An experiment was made in which sample *G* (Table III), a hypereutectic silicon iron containing 0·83% of phosphorus was heated to 1050° C., soaked for 4 hr., and quenched in water. The resultant microstructure shown in Fig. 12, contained no dispersed phase. On annealing the specimen at 1050° C. for 4 hr. a very definite dispersed phase appeared in the alpha grains, Fig. 13. An exactly similar phenomenon occurred when hypo-eutectic silicon-iron samples *H* and *I* were treated in the same way.

The production of the dispersed phase on annealing the water-quenched alloy could be accomplished at 800° as well as at 1050° C., but the attainment of the higher temperature was necessary to absorb into the alpha phase the dispersed phase in the first instance. The extreme minuteness of the dispersed phase rendered its identification difficult. An experiment was made in an effort to cause the dispersed particles to agglomerate under the influence of long annealing. Figure 14 records the result of a 3 weeks' annealing treatment at 800° C. carried out on sample *L*, Table III. The numerous fine particles, visible in the alpha phase in Fig. 12, agglomerated into a relatively few spheroidal inclusions scattered throughout the alpha and epsilon phases. These spheroids were similar in every respect to the massive phosphide phase also present. It is interesting to note that the eta phase originally present in this specimen was decomposed and that only the alpha and epsilon phases remained after annealing. The annealing experiment just described would appear to indicate that the dispersed phase found in all sand-cast silicon-iron alloys with over 0·1% of phosphorus, is a phosphide precipitated in the solid state during cooling.

TABLE IV—*Compositions of Specimens of 14–15%-Silicon Iron Containing Manganese*

Sample	Method of Melting	Manganese, %	Silicon, %	Carbon, %	Phosphorus, %
A	Arc furnace	0·53	15·2	0·50	...
B	Arc furnace	1·80	14·4	0·67	0·08
C	High-frequency furnace	2·53	14·6	0·56	0·07
D	High-frequency furnace	3·70	14·6	0·54	0·05
E	Arc furnace	7·30	13·6	0·68	0·07

THE INFLUENCE OF MANGANESE ON THE MICROSTRUCTURE

Specimens of commercial silicon iron containing 0·5–7·3% of manganese were cast for metallographic examination in the manner described in the previous section. The compositions of the experimental melts are listed in Table IV. The microscopical examination was carried out on sections cut from sand-cast ½-in. dia. test bars.

Manganese additions up to 2% had little effect upon the microstructure of the sand-cast alloy ; the addition was apparently taken up in the silico-ferrite. There was a greater carbide stability noted when the alloy was chill cast. As the manganese was increased above 2%, more of the available carbon in the iron was found to exist in the combined state, even in sand castings. With 7·5% of manganese in the iron, about half the carbon was present as carbide, and the remainder existed as a 'fine graphite' which is known to be the first decomposition product of the boundary carbide in acid-resisting silicon irons.[6] Figure 15 shows at ×600 diameters the

microstructure of alloy *E*, Table IV, which contains 7·3% of manganese. It will be noted that in this photomicrograph, as in the microstructures of all the other manganese-containing samples examined in this section, no dispersed phase was present in the alpha grains.

The Influence of Molybdenum on the Microstructure

An alloy containing 15% of silicon and 3–4% of molybdenum is well known to possess improved resistance to corrosion by hot hydrochloric acid and is thus of interest commercially. An examination of the microstructure of silicon iron containing molybdenum has been made, using alloys prepared in a laboratory high-frequency furnace and cast in the manner described in previous sections into ½-in. diameter test bars. The chemical composition of the specimens is given in Table V.

Table V—*Compositions of Specimens of 14–15%-Silicon Iron Containing Molybdenum*

Sample	Method of Melting	Molybdenum, %	Silicon, %	Carbon, %	Manganese, %	Phosphorus, %
A	High-frequency furnace ...	3·50	14·3	0·65	0·50	0·052
B	High-frequency furnace ...	3·04	14·1	0·59	0·40	0·111

The addition of 3½% of molybdenum caused the development of a new phase having the appearance of a eutectic. There was a complete absence of graphite, indicating the retention of carbon as a carbide, very probably in association with molybdenum. The microstructure of specimen *A*, Table V, containing 3½% of molybdenum, is shown at ×600 and ×1200 diameters in Figs. 16 and 17, respectively.

The complex carbide phase showing in Fig. 17 proved to be very stable; annealing at 1000° C. for 4 hr. failed to cause any change in the microstructure. The carbide which has been noted in ordinary unalloyed 14–15% silicon iron, under conditions of rapid cooling from the molten state,[6] breaks down readily after a short anneal at 650° C. There is little doubt, therefore, of the stabilizing influence of molybdenum upon the carbide shown in Figs. 16 and 17.

Observations on a second molybdenum-containing silicon iron, sample *B*, Table V, make it clear that in order completely to stabilize the carbides a percentage of between 3·0% and 3·5% of molybdenum is required. In addition to complex carbides, a residual amount of carbon as acicular graphite was recorded in this specimen, which contained 3·04% of molybdenum. Annealing at 1000° C. did not cause any variation in the quantity or disposition of the phases.

The Influence of Copper on the Microstructure

For the study of the influence of copper additions on the 14–15% silicon iron, a series of alloys of compositions assembled in Table VI were prepared in the laboratory high-frequency furnace. As in the other experimental work recorded in this paper, the micrographical examination was made chiefly on ½-in. dia. sand-cast bars poured at temperatures between 1450° and 1500° C. (measured by optical pyrometer).

Table VI—*Composition of Specimens of 14–15%-Silicon Iron Containing Copper; Melted in High-Frequency Furnace*

Sample	Copper, %	Silicon, %	Carbon, %	Manganese, %	Phosphorus, %
A	0·56	13·6	0·50	0·44	0·062
B	1·26	14·0	0·48	0·57	0·060
C	2·12	14·6	0·58	0·52	0·066
E	3·32	14·2	0·44	0·50	0·054
G	4·68	15·1	0·42	0·47	0·057
I	7·80	14·0	0·40	0·54	0·080

The first influence of copper on the microstructure of 14–15% silicon iron was the appearance of a dispersed phase in the alpha silico-ferrite areas, which occurred when only 0·56% of copper was present in the sample. This dispersed phase which, in the specimen sample *A*, Table VI, was very similar to the spot-like markings in the phosphorus-containing silicon irons, is shown in

Fig. 18. With $1 \cdot 26\%$ of added copper (sample *B*, Table VI, in the sand-cast condition) the test piece showed some residual carbide with which was associated in isolated patches a very small amount of free copper or a copper-rich phase. The carbide was decomposed on annealing at 800° C. and the golden copper-rich phase remained. With sample *C*, Table VI, having $2 \cdot 12\%$ of copper, appreciable amounts of this copper-rich phase appeared, usually occupying a position close to patches of the acicular variety of graphite. This characteristic persisted throughout the entire range of copper additions, and with $7 \cdot 80\%$ of copper, sample *I*, Table VI, the graphite became entirely of a very fine acicular type, Fig. 23.

The dispersed phase found in sand-cast commercial copper–silicon–iron alloys responded to heat-treatment in almost the same way as did the dispersed phase produced by phosphorus additions. The sand-cast $2 \cdot 12\%$ copper alloy, sample *C*, Table VI, showed, as in Fig. 19, the dispersed phase in the alpha areas and small inclusions of a massive copper-rich phase at the grain boundaries. On heating to 1050° C. for 4 hr. and subsequently quenching the specimen in water, the alpha grains were found to be almost free from dispersed phase, and the inclusions at the alpha-phase boundaries were very much reduced in size and number, Fig. 20. Slow cooling from 1050° C. gave an abundance of the fine phase and also more of the massive copper-rich phase as in Fig. 21. When the larger spots in the annealed specimen were examined under oil immersion at high magnification it was possible to observe in the microstructure that they had a faint copper colour, and it is thought that they are a part of the copper-rich phase which is precipitated from the alpha solid solution on slow cooling.

The changes which took place in the type of graphite when additions of copper were made to commercial acid-resisting silicon-iron were very interesting; the flake type of graphite was gradually displaced by the rod-like acicular form. Figure 22 shows vividly that copper plays an important part in this transformation. The globules of copper-rich phase tended to deposit preferentially along the length of the graphite needles, which in this case were arranged in a very definite 60° Widmanstätten pattern.

The Influence of Residual Elements

The principal residual elements observed in commercial acid-resisting silicon-iron alloys are titanium, aluminium, and sometimes nickel. For the most part they arise from the commercial grades of ferrosilicon used. Titanium has been encountered in amounts up to $0 \cdot 35\%$ and in the micro-examination of such specimens the characteristic cubic crystals of titanium carbide can be observed. Aluminium is present in practically all commercial silicon irons in small amounts, and percentages as much as $0 \cdot 05\%$ have been encountered. Nickel has been met with in certain commercial alloys, and in one case has been traced to the presence of nickel in the original ferrosilicon. In the case of both aluminium and nickel no separately identifiable phase has been observed under the microscope.

General Remarks

This study of the microstructure of commercial acid-resisting silicon-iron alloys has had the advantage of having been carried out alongside work on the X-ray examination of the system. It is of considerable importance to note that the existence of phases revealed by X-ray crystallographic methods can only be confirmed with great difficulty by ordinary metallographic methods and, in fact, in the case of α'', ordinary metallographic methods so far have failed to confirm the findings of the X-ray crystallograph.

The information reported previously on the metallographic features of these alloys is very scanty and has frequently been misleading, and for this reason it is hoped that this careful study will be of value.

Acknowledgments

The work described was carried out in the laboratories of the Ministry of Supply Silicon Iron Factory and under the ægis of the Silicon Iron Advisory Panel, of which Dr. W. G. Hiscock is Chairman. The X-ray investigations, to which reference is made, were carried out at the Cavendish Laboratory under the direction of Sir Lawrence Bragg.

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FIG. 18—The dispersed phase in a commercial silicon iron containing 0·56% of copper; specimen *A*, Table VI × 600

FIG. 19—Massive copper-rich phase and dispersed phase in sand-cast silicon-iron alloy containing 2·12% of copper; specimen *C*, Table VI × 600

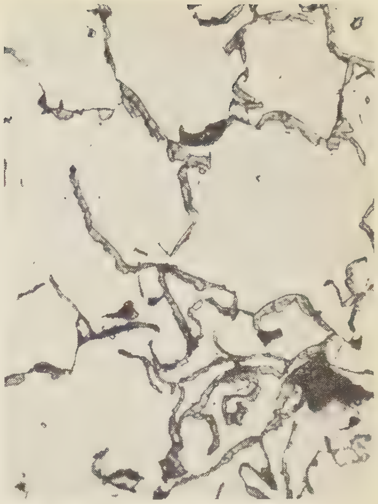

FIG. 20—Same sample as in Fig. 19, but heated to 1050° C. and quenched in water. Dispersed phase and massive copper-rich compound almost absent × 600

FIG. 21—Same sample as in Fig. 19, slowly cooled from 1050° C. Dispersed phase and massive copper-rich compound again present × 600

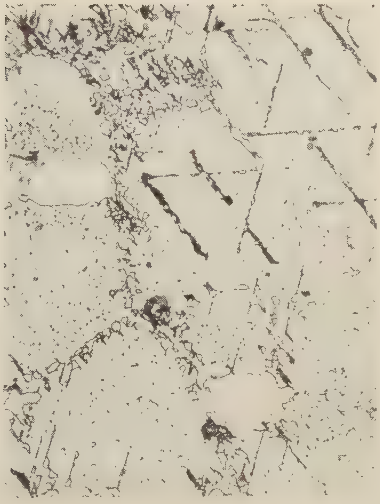

FIG. 22—Specimen *G*, Table VI, containing 4·68% of copper. Note the deposition of copper along the rodlike or acicular form of graphite × 600

FIG. 23—The very fine acicular type of graphite in close association with massive copper-rich phase in specimen *I*, Table VI, containing 7·80% of copper × 600

The microsections appearing in Figs. 18–23 were etched in the nitro-hydrofluoric acid reagent
(Figs. 18–23 were reduced to four-fifths linear in reproduction)

The Application of Electrolytic Polishing to Ferrous Metallography*

By H. J. Merchant†

SYNOPSIS

Preliminary experiments on electrolytic polishing were conducted to determine the effects of variable conditions of electrolysis when using Jacquet's perchloric acid/acetic anhydride electrolyte. The design of a suitable type of cell was also investigated and five different cells were constructed. Electrolytic polishing is compared with mechanical polishing on the basis of procedure and results obtained; specimens containing the graphite phase, large amounts of slag, or discontinuities, do not respond to electrolytic polishing. Other potential solutions for electrolytic polishing were also investigated, and from these a list of recommended electrolytes is given. The most useful acids for metallographic purposes are perchloric acid, nitric acid, and chromic acid, diluted where possible with acetic anhydride, acetic acid, or alcohol. Electrolytes based on phosphoric acid or sulphuric acid cannot be recommended for metallographic polishing owing to their tendency to produce pitting or polish attack. Various observations made in the course of this work are then considered relative to the present theory of electrolytic polishing.

INTRODUCTION

ELECTROLYTIC polishing is a comparatively recently discovered phenomenon; the first mention[1] of it was made in 1925, and most development work on it dates from 1935. Since then, the phenomenon has received much study from Jacquet in France and also from various American investigators.[2] The possibility of replacing the tedious procedure of mechanical polishing by a simple electrolytic method has had a universal appeal, but unfortunately, not a universal following, and it was considered that some research would be useful in assessing the relative merits of both methods. Mechanical polishing involves the use of a developed technique and skill on the part of the operator, but electrolytic polishing is such that once the technique has been evolved the element of skill is unnecessary, and also, polishing is effected in a shorter time.

Electrolytic polishing produces a smooth and brilliant surface by the selective removal of metal from the surface by anodic action. The specimen is made, or attached to, the anode of an electrolytic cell, the cathode usually being a simple plate of a similar metal to that being polished. Essentially, the apparatus consists of a vessel enclosed in a water jacket, the anode consisting of or containing the specimen, the cathode, a stirrer, a thermometer, and a variable D.C. supply connected in series with an ammeter and the electrodes of the cell. A voltmeter across the electrodes may prove useful but is not essential. A simplified sketch of the apparatus is shown in Fig. 1. Electrolytes must be of selected and controlled composition. Various factors can affect the process and prevent successful polishing, and these will now be more fully discussed.

PRELIMINARY INVESTIGATIONS

With the object of investigating the various operational factors which affect the results obtained in electrolytic polishing, preliminary investigations were conducted using as an electrolyte Jacquet's perchloric acid/acetic anhydride solution. This electrolyte appeared most suitable for this purpose; it will be referred to in this work as Jacquet's solution. The electrolytic polishing method also seemed to lack a convenient type of cell, and this problem was considered first.

Type of Cell Used

In an electrolytic polishing cell the anode connection for transmitting the current to the specimen constitutes the most difficult problem. It is desirable that the area of the anode clamp or connection exposed to the electrolyte should be as small as possible, preferably much smaller than the specimen area. Other features of the cell for successful polishing may be listed as follows:

(1) The anode connection should be constructed so that the specimen may be removed from the cell easily and quickly for washing and subsequent treatment;

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* A report received 2nd August, 1946, on a research carried out with the aid of a grant from the Andrew Carnegie Research Fund.

† Commercial Steels and Forge Co., Ltd., Sydney, Australia.

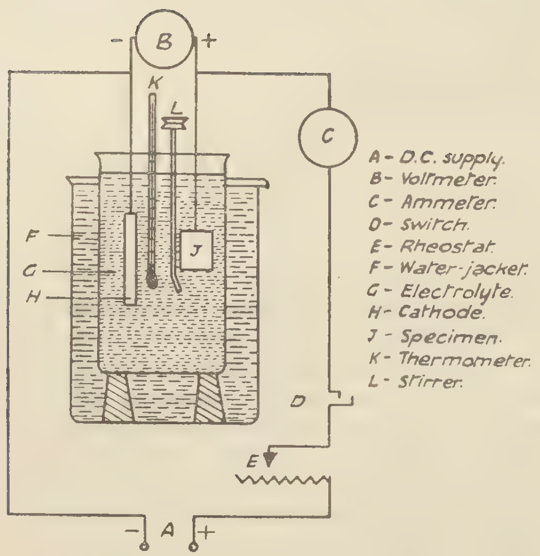

FIG. 1—Essential apparatus for electrolytic polishing

it should also preferably be made of corrosion-resisting material so as to give a reasonable life.

(2) The specimen should not be in contact with the electrolyte except when the required potential difference for polishing exists. This requires that the current must be switched on before the specimen is immersed in the electrolyte and that the specimen should be removed from the electrolyte and quickly washed before the current is switched off.

(3) The current-density on the specimen must be accurately controlled.

(4) The position of the specimen with respect to the cathode during electrolysis should remain fixed, so that no unnecessary variable of internal resistance of the cell will be introduced.

(5) The cell should permit some agitation of the electrolyte.

(6) The temperature of the electrolyte should be easily controlled and preferably kept constant during electrolysis.

(7) The volume of the electrolyte should be such as to permit free agitation and conduction of heat to the cooling medium.

(8) The cathode must not react chemically with the electrolyte.

In these preliminary experiments five types of cell were constructed and tried. The wooden-box type of cell[3] proposed in the U.S.A. was tried out but was found to be unsatisfactory for the general requirements of a metallographic laboratory. Four other types of cell were devised, all similar except in the design of the anode clamp. In each case the vessel containing the electrolyte was a litre beaker contained in a larger beaker which served as a water jacket. A stirrer was finally adopted as essential to the dispersion of the heat generated in the cell and consequent uniform polishing. A thermometer with its bulb adjacent to the specimen was employed. A large volume of electrolyte was found to be useful in absorbing the heat evolved in the cell and in not ageing too quickly. The cathode in all types of cell was made of stainless steel sheet.

(1) *The Screw-Clip Type of Polishing Head*

This is illustrated in Fig. 2. The specimen is located in the anode clamp which should be at a fixed distance from the cathode. The distance should not be too large, otherwise a high voltage is required to obtain the correct current-density; nor too small, as local heating may develop and the current may even flash over from one electrode to the other; 5 cm. is suggested as a reasonable maximum distance and 5 mm. as a

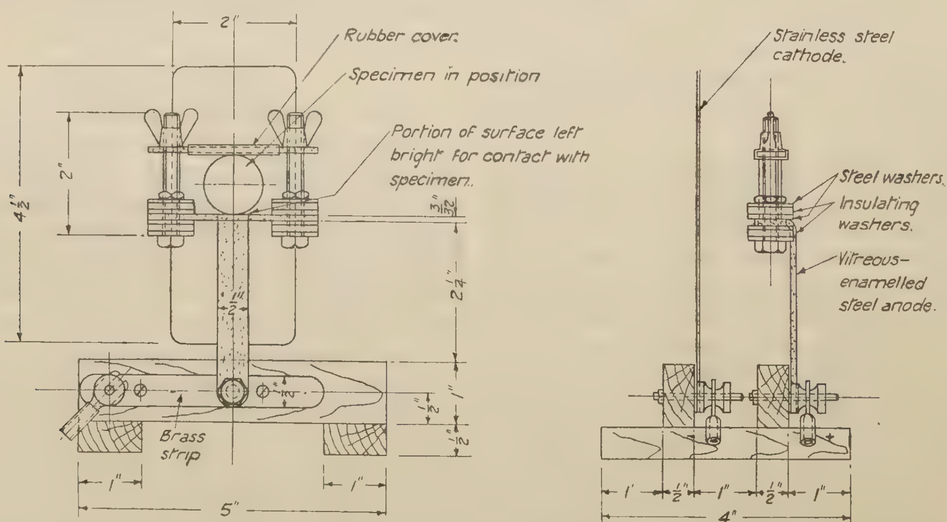

FIG. 2—Inverted views of screw-clip type of polishing head, complete with specimen

safe minimum distance. The anode clamp holds the specimen firmly, the latter being well immersed in the electrolyte. For ferrous specimens, the clamp must be made of a ferrous metal. All but that part of the clamp contacting the specimen was insulated from the electrolyte, and the solution to this problem was found by vitreous-enamelling all except the exposed contact area of approx. 3 sq. cm. This is small compared with the surface area of the average microspecimen (10–20 sq. cm.). In use, the contact area gradually polishes away and the clamp must eventually be replaced. Insulation of the clamp is necessary, otherwise the total current necessary to obtain the correct current-density on the specimen is large and consequently much heat is generated in the cell. For successful enamelling, the clamp should be made of low-carbon steel sheet with an ample radius at all corners. The screws and wing nuts of the clamp presented a problem which was eventually solved by insulating them with porcelain or rubber sleeves with washers at the points of location in the clamp ; it was also found useful to have the movable member of the clamp covered with rubber tubing, so as to avoid damage by the specimens to the enamel coating.

(2) *The Horizontal-Platform Type of Polishing Head*

This type of polishing head is illustrated in Fig. 3, and consists essentially of a horizontal anode platform on which the specimen is placed, the cathode being located horizontally above the platform. Here also, most of the anode except the contact area (about 3 cm. sq.) was insulated by vitreous-enamelling. The advantages of this type of polishing head are that (i) no clamps are required to hold the specimen, hence ease and speed of operation ; (ii) a constant current may be applied providing the specimen area is not too large compared with that of the contact area ; and that (iii) two or more small specimens may be polished at the same time. The disadvantages are that (i) contact with the specimen is not too good, it being dependent only on the mass and shape of the specimen, hence the required current-density may not be obtained ; and that (ii) some degree of care is necessary in lowering the polishing head into the solution. A similar type of cell has been evolved by Lowgren and Hildebrand,[4] who claim that increased polarization results from a horizontal specimen surface. The author, however, has found it essential to agitate the electrolyte, and in this case similar results are obtained from vertical or horizontal surfaces.

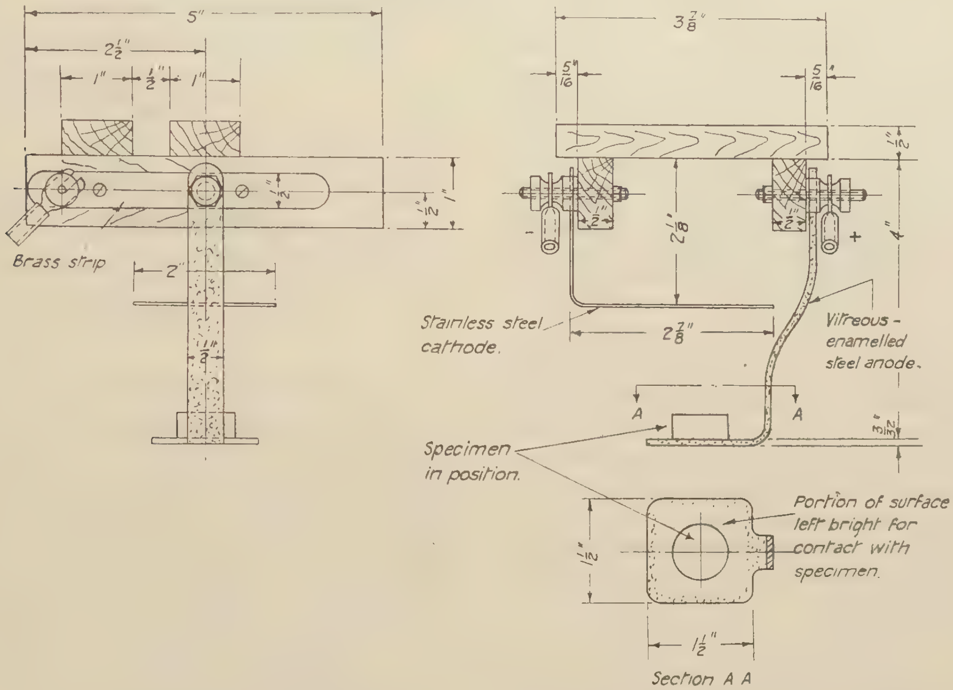

FIG. 3—Upright views of horizontal-platform type of polishing head, complete with specimen

(3) *Polishing Head Used for Investigation of Potential Electrolytes*

This type is shown in Fig. 4, and was designed to offer simplicity and speed of operation necessary to investigate many different potential electrolytes and polishing conditions. No anode clamp is used, and the specimen is connected to

the bus-bar by means of a $\frac{1}{4}$-in.-dia. steel rod, which may be insulated from the electrolyte by vitreous-enamelling or by rubber tubing. The specimen is attached to the rod by drilling and tapping it with a $\frac{1}{4}$-in.-dia. threaded hole into which the rod is fitted, the latter being connected to the bus-bar by an adaptor, making it possible to remove the specimen and rod from the polishing magnet by contact with the anode bus-bar, which in this instance is of steel, and thence to the specimen by means of magnetic contact with it. This type of polishing head affords rapid operation and irregularly shaped specimens offer no difficulties, but it is not of course suitable for non-magnetic specimens such as austenitic steels, and it is necessary to adjust the height of the head

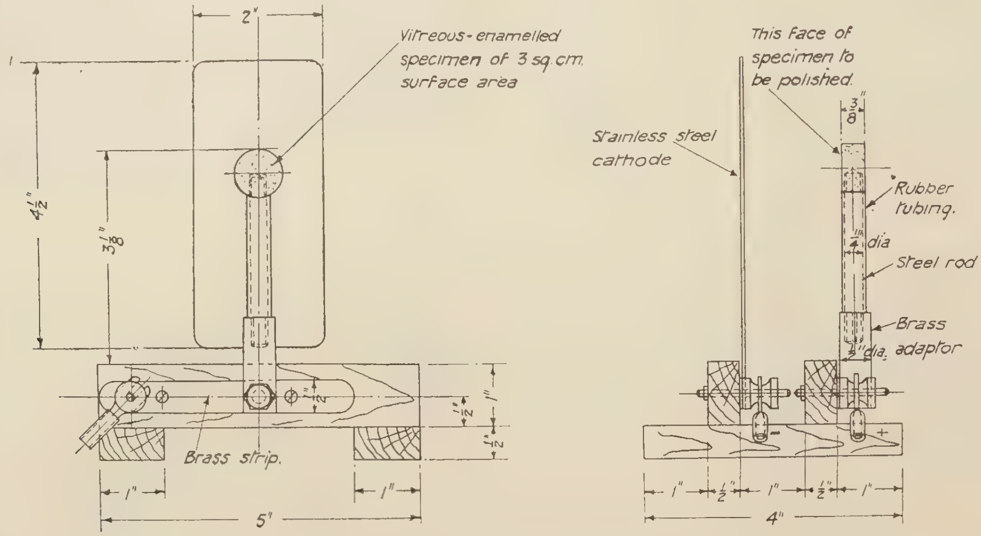

FIG. 4—Inverted views of polishing head used for the investigation of potential electrolytes

head very quickly. A further feature of this cell was that the specimen was enamelled all over except on the surface to be polished, with the advantage that an accurately known plane faced the cathode. This procedure is not suitable for routine work, but it served well for research work on different electrolytes.

(4) *Magnet-type Polishing Head*

This type of anode connection employs a short powerful bar magnet suspended from a rigid cross-bar arrangement similar to that used in other heads, so that the specimen when held by the magnet is just immersed in the electrolyte, a horizontal cathode being employed underneath the specimen. The potential is transferred to the

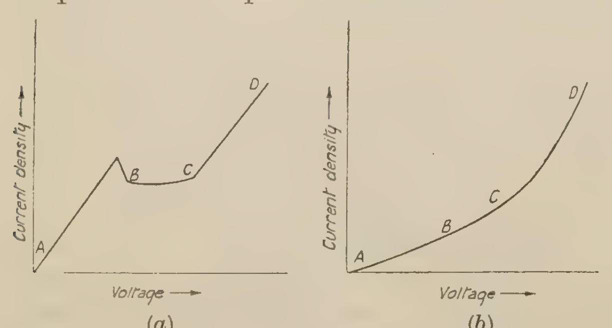

FIG. 5—Polishing curve for metals of (*a*) the first group, *e.g.*, copper, cobalt, and zinc ; and (*b*) the second group, *e.g.*, aluminium, tin, iron, lead, and nickel

so that only the specimen and not the magnet is immersed.

Effect of Variable Conditions of Electrolysis

Work was undertaken to investigate the variable factors in electrolytic polishing, which may be listed as follows :

(*a*) Potential difference or voltage applied.
(*b*) Current-density applied.
(*c*) Current-density distribution on specimen.
(*d*) Temperature of the electrolyte.
(*e*) Agitation of the electrolyte.
(*f*) Time of polishing.
(*g*) Initial preparation of the specimen.
(*h*) Type of cell used.
(*i*) Age of the electrolyte.

(*a*) *Potential Difference or Voltage Applied*

Jacquet[5] has divided the metals and alloys for electrolytic polishing purposes into two groups according to the essential factor controlling the electrolysis, namely, either (i) the terminal potential of the cell, or (ii) the current which traverses it. In the first group are such metals as copper, cobalt, zinc, and alloys thereof, and here the potential difference between the anode and cathode is the controlling factor. In the second group are such metals as aluminium, tin, lead, iron, and nickel, and certain alloys thereof, and in this group the current-density is the factor which must be controlled. The different response during electrolysis may be best illustrated by the rough

current-density/voltage curves for the two groups of metals, which are shown in Fig. 5. The first group polish in electrolytes of fairly high electrical conductivity, whereas the second group only polish satisfactorily in electrolytes of fairly low electrical conductivity. Iron may be polished with difficulty in high-conductivity electrolytes,[6] but the most useful results are obtained with low-conductivity electrolytes such as Jacquet's solution. Such electrolytes require a relatively high voltage for operation, but the current-density alone governs the polishing conditions. A suitable source of current for electrolytic polishing is a rectifier giving an open-circuit voltage of 100 v. and up to 30 amp. full-load current, with voltage regulation in easy stages.

(*b*) *Current-Density Applied*

Using Jacquet's solution and a mild-steel specimen the effect of current-density on the polishing process was investigated, all other conditions being kept constant. A moderate agitation was employed; surface preparation was standardized using a 000 emery-paper finish, and the polishing time was 10 min. The following observations were made :

Current-density, amp./sq. cm.	Observations
0·01 ...	Specimen did not polish but became pitted and etched.
0·02–0·04 ...	Specimen became polished, and on etching the micro-constituents were well developed, but the structure incorporated signs of a defect which may be termed "polish attack."
0·04–0·10 ...	Specimen became progressively more polished as the current-density was increased, with good etching characteristics.
0·10–0·20 ...	Specimen became polished, but the metallographic response was not so good.

Subsequent trials at current-densities up to 0·20 amp./sq. cm. with shorter times, however, gave satisfactory results. At all values above 0·02 amp./sq. cm., a viscous brown film formed on the specimen. These preliminary trials showed that (1) polarization, indicated by falling current values, took place rapidly, necessitating a voltage adjustment to maintain the current-density constant; (2) the range of current-density in which optimum polishing occurs is fairly narrow, and that (3) increasing the current-density decreases the polishing time.

(*c*) *Current-Density Distribution on Specimen*

In the above experiments, a cylindrical mild-steel specimen 2 cm. dia. and nearly 1 cm. high with rounded corners was used, the approximate over-all area being 12 sq. cm. It was observed however, that the sides and the face of the specimen remote from the cathode were not polished to the same degree as the surface facing the cathode, and it was also noticed that poor contact of the specimen with the anode clamp resulted in uneven current-density distribution. This lack of evenness may be termed 'covering power,' as it is somewhat analogous to the term 'throwing power' used in electrodeposition. In investigating the effects of current-density distribution the polishing head illustrated in Fig. 4 was used, and runs were made with an ordinary specimen of 12 sq. cm. in area, and also with the same specimen enamelled all over except for the polished surface of 3 sq. cm. in area which faced the cathode. A comparison of results with the same calculated current-density showed that no significant difference could be detected visually or metallographically on the polished surface. It may therefore be concluded that with an ordinary specimen, even though the sides and surface remote from the cathode may not be polished, the removal of metal on the surface being polished is such that the desired result is obtained when the current-density is calculated on the whole surface of the specimen. Usually only one face of a specimen is required to be polished, and the actual current-density on that face depends on its distribution over all the specimen. The enamelled specimen approaches the ideal in obtaining a well defined current-density, but subsequent trials with electrolytes of poor covering power indicated that even this was not perfect, a brightly polished annular ring on the specimen, surrounding an unpolished centre, being apparent at some current-densities.

In calculating the current for a clamp or 'grip' of thin sheets made into a single specimen it is not advisable to take into account the area of each sheet. In polishing cross-sections of tubes, however, it would be advisable to allow for the external and internal surface areas. The exposed areas of clamps used for holding awkward specimens must also be considered when assessing the current required.

One aspect of current-density distribution in electrolytic polishing is an adverse one, inasmuch that a local increase of current-density occurs at the edges and corners of specimens, also at the location of any cracks or discontinuities. The effect is that more metal is removed at such points of high current-density, resulting in the locality being out of true metallographic plane. Furthermore, the increase in current-density occurring at such localities may be such that polishing does not take place there under optimum conditions, and local pitting may occur. When

it is desired to examine a specimen right up to the edge, the iron plating method used by Jenkinson[7] is the most satisfactory means of obtaining a uniform polish. Generally, however, electrolytic polishing cannot be successfully employed where it is desired to examine the adhesion of surface coatings such as zinc, tin, lead, etc., on steel or for the examination of surface cracks or defects.

(d) Temperature of the Electrolyte

With Jacquet's solution, optimum results are obtained when the temperature is kept at 15–20° C. and is not allowed to exceed 25° C. Increase of the electrolyte temperature increases the conductivity and lowers the voltage required to give the same current-density. Experimental trials showed that at increasing temperatures between 15° and 30° C., the quality of the polish progressively deteriorated at the normal current-density, and was only restored by increasing the latter to re-establish conditions for polarization and the formation of the anodic film. Over 30° C., an etching action and a resultant dark carbonaceous film appeared instead of the brown anodic film. In addition, all electrolytes containing acetic anhydride or acetic acid gave rise to obnoxious fumes when operating over 30° C. Operating temperatures of 15°–20° C. are ideal, making it very convenient to cool the vessel containing the electrolyte with mains water. A low temperature is advantageous in that the viscosity of the electrolyte is kept high, thus favouring increased covering power. Temperature uniformity of the electrolyte is important in obtaining uniform polishing results, and in this respect a moderate agitation of the electrolyte aids the dispersion of the heat evolved in the anode–cathode area.

(e) Agitation of the Electrolyte

Using Jacquet's solution with all other conditions standardized at the optimum for best results, trials were made to determine the effect of agitation on the polishing process. Results showed that although a satisfactory polish can be obtained in a still electrolyte, a moderate agitation is a definite advantage. Without agitation the temperature of the electrolyte in the anode–cathode area increases rapidly, resulting in an increase in conductivity and current-density which may give erratic results. Agitation does not increase the time required for polishing.

(f) Time of Polishing

The time required for electrolytic polishing depends on the current-density and the initial surface preparation. The higher the current-density which will affect polishing, the less time required. There is, however, a definite amount of metal to be removed to effect polishing and this depends on the surface preparation. If insufficient metal is removed, the surface will retain such characteristics as residual scratches ; if polishing proceeds after the optimum stage, the surface may obtain some undesirable characteristics, such as undue attack on the non-metallic inclusions or preferential attack of some constituents.

For investigating the time required for electrolytic polishing, specimens were prepared on several grades of emery paper and then electrolytically polished to give the optimum polish when examined visually, and also when etched and the structure examined microscopically. Specimens were also fully mechanically polished using a suspension of chromic oxide in water. The results indicated that the time required for polishing decreases with increasing fineness of initial surface preparation. The following results were obtained using Jacquet's solution and a current-density of 0·04 amp./sq. cm. :

Initial surface finish	Time required, min.
0 Emery paper	16
00 „ „	10
000 „ „	8
0000 „ „	7
Wet chromic oxide	3

For a constant current-density or current on any given specimen, the same mass of metal is removed from the specimen in the same time, irrespective of the degree of surface preparation. A longer polishing time is required, however, when a coarser finish is employed, because more metal must be removed anodically to erase the coarser elevations and produce a plane surface, and for any given current-density there is a minimum time required to erase all the scratches and effect polishing.

(g) Initial Preparation of the Specimen

The initial surface preparation of the specimen is important, especially where polishing times are of the order of minutes and not seconds. Anodic brightening takes place whatever the surface finish, but unless this is relatively smooth a perfect result will not be obtained ; furthermore, unless the surface is such that polishing takes place in a reasonable time, over-polishing of parts of the specimen (*e.g.*, around inclusions, defects, or regions of high local current-density) will occur. In order to study the effect of initial surface preparation, specimens were prepared on the common grades of emery paper and subsequently electrolytically polished. Two types of specimens were employed, one being of 0·40% carbon steel in the annealed condition, and the other being of the same steel in the hardened condition ; each specimen took the form of a cylinder

2 cm. in dia. and 1 cm. high with rounded corners, thus having a total surface area of approximately 12 sq. cm. and a polished surface area of approximately 3 sq. cm. Jacquet's solution with a current-density of 0·04 amp./sq. cm. was used. Before and after both the initial preparatory mechanical polishing and the subsequent electrolytic polishing operation, the specimens were weighed and the approximate average thickness of metal removed was calculated. The object of these measurements of the metal removed in preparatory polishing was to measure roughly the depth of ' cut ' or finish put on the specimen by different grades of emery paper. Polishing on any paper was carried out until the scratches previously made by the same grade of paper were erased by polishing at 90° to the previous direction. In preparing the specimens on emery paper, care was taken to give the same treatment, standardizing the number of strokes and the pressure, and using a new section of paper at each trial. Specimens were also fully mechanically polished for a standard time on a rotating wheel impregnated with chromic oxide and water. In addition, the specimens were weighed before and after etching, both after mechanical polishing and electrolytic polishing. The results obtained are shown in Table I, and are the average of six trials on both types of specimens. They are conclusive in that they do show a diminution of the thickness of the metal removed, ranging from approximately 8μ for a surface prepared on 0 emery paper to approximately 1·5μ for a surface fully mechanically polished. The hard specimen showed less diminution of weight than the soft specimen, indicating its greater abrasive resistance.

With Jacquet's solution it was found that with a 000 emery-paper finish, 0·04 amp./sq. cm. and 8 min. polishing time were the minimum conditions for a complete scratch removal in electrolytic polishing, and under these conditions the experiments showed that the average thickness of metal removed in polishing was 6μ. This figure is in fair agreement with that of 4μ obtained for the average 'thickness' of the '000' preliminary polish employed. The quantity of iron dissolved anodically in the form of bivalent ions Fe^{++} should be 0·695 mg./sq. cm./min. with a current-density of 0·04 amp./sq. cm., assuming no loss of energy in the cell. The figure of 0·59 mg./sq. cm./min. obtained for this value in practice represents an efficiency of the cell of about 85%.

TABLE I—*Rough Measurements of Metal Removed in various Polishing and Etching Procedures*

Procedure	Metal Loss on Specimen, mg.		Metal Loss, mg./sq. cm.		Average Thickness Removed, mm.		Approximate Average Thickness Removed in Procedure, μ
	Hard	Soft	Hard	Soft	Hard	Soft	
Normal hand polishing on 0 emery paper	18	20	6·0	6·7	0·0077	0·0086	8
Normal hand polishing on 00 emery paper	13	17	4·3	5·7	0·0055	0·0073	6
Normal hand polishing on 000 emery paper	9	10	3·0	3·3	0·0038	0·0042	4
Normal hand polishing on 0000 emery paper	6·5	7·5	2·2	2·5	0·0028	0·0032	3
Normal polishing on rotating wheel with chromic oxide and water ...	3·8	4·0	1·3	1·3	0·0017	0·0017	1·5
5 sec. etch in 2% nital after electrolytic polishing	0·7	0·8	0·06	0·07	0·00008	0·00009	0·10
15 sec. etch in 2% nital after orthodox mechanical polishing	1·8	3·0	0·15	0·25	0·00019	0·00032	0·5
	Average						
Electrolytic polishing with Jacquet's solution under optimum conditions (' 000 ' finish, 0·04 amp./sq. cm., 8 min. polishing time)... ...	57·0		4·75		0·0061		6
Rate per min., under optimum conditions	7·1		0·59		0·00076		0·75

(*h*) *Type of Cell Used*

The effect of such variables as the anode and cathode shape, size, and position was explored. The anode shape and size is more or less controlled by the specimen characteristics, but where anodic clamps are used their immersed area should be kept at a minimum by insulation. The contact with the specimen should be as good as possible, and the surface to be polished should face the cathode. Wherever possible small specimens should be used in order to minimize heat generation, and also consequent early ageing of the electrolyte. The average microspecimen is usually from 10 to 20 sq. cm. in surface area, and this can be satisfactorily polished in a litre of electrolyte. Equally satisfactory results were obtained with the anode surfaces both vertical and horizontal, the latter position being tried when the specimen surface faced both up and downwards.

Investigation of the size, shape, and position of the cathode showed that a large cathode is desirable, as it gives a more even distribution of current-density on the specimen and also a more even distribution of heat generated during electrolysis. A cathode area of at least 50 sq. cm. is recommended. For ferrous specimens, either iron or aluminium is a suitable metal for the cathode, but one made of stainless-steel sheet is recommended owing to its corrosion resistance. The cathode position, whether vertical or horizontal, is immaterial, except that it should face the specimen surface and preferably ' cover ' it well in location.

(*i*) *Age of the Electrolyte*

Observations were made on the life of Jacquet's solution and they apply in general respects to other electrolytes. When prepared, the electrolyte has a specific gravity of 1·160–1·165 g./c.c. but in use the electrolyte becomes charged with iron which increases its density. This proceeds with little effect on the polishing power until a specific gravity of 1·195 g./c.c. is reached, after which the potential polishing power falls off rapidly. Some anodic brightening occurs but this is inevitably accompanied by "polish attack," indicating a current-density slightly too low for optimum results. Some extended use may be made of the electrolyte by increasing the current-density, or by adding small quantities of water of not more than 1% at any one time, and allowing the electrolyte to stand for some hours before further use ; if more water is added, the potential polishing power of the electrolyte is destroyed. When the specific gravity exceeds 1·195 g./c.c., it is recommended that the electrolyte be discarded and a new one prepared. When in use electrolytes should be protected from contamination with water, and when not being used precautions should be taken to prevent evaporation and consequent change of composition.

Comparison with Mechanical Polishing

In order to compare electrolytic polishing with mechanical polishing, both the process entailed and the results obtained must be considered. As far as the process is concerned, the following conclusions arose from this work :

(1) Electrolytic polishing is quicker than mechanical polishing. Electrolytes have been developed which will polish satisfactorily in 15 sec. or even less. With Jacquet's solution polishing takes only a few minutes, comparing very favourably with mechanical polishing, which may take up to 30 min. to produce a satisfactory surface. Large specimens can be polished as quickly as small specimens.

(2) The type of personal skill needed for mechanical polishing is not required in electrolytic polishing, and once the conditions of electrolysis have been determined, subsequent operation becomes a matter of easy routine. Depending on the time required for electrolysis, the operation may be left temporarily and other work performed, as for example, preparation of the next specimen.

(3) From a consideration of the saving of time, labour, and polishing materials, electrolytic polishing is cheaper than mechanical polishing.

(4) The initial surface preparation before electrolytic polishing need not be so carefully performed as in the case of mechanical polishing, especially when electrolytes are used which permit high current-densities.

(5) It has been reported that different electrolysis conditions are required for different alloys and tempers, but this has not been the author's experience in polishing a wide range of ferrous specimens once the desired optimum conditions have been obtained. The only variables necessary are the current-density and the resultant time required for polishing. Generally, a wide range of carbon and alloy steels, except those containing free graphite or large quantities of non-metallic elements, may be polished at the same current-density ; austenitic steels, however, respond better at current-densities about 50% higher than those used for ordinary steels.

The results obtained by electrolytic polishing compare favourably with those obtained on surfaces mechanically polished. Visually, an electrolytic polish is somewhat brighter than that possible with the most careful mechanical polishing. This has been proved by Jacquet[8] in the case of copper, but it is easily observable after some experience in polishing steel. It has been

stated that an electropolished surface has a wavy finish, and is not optically flat; this, however, need not interfere with the examination of specimens at magnifications usefully employed for ferrous metals, *i.e.*, from 50 to 2000 diameters.

A steel specimen which has been polished electrolytically reveals on examination a slight attack on the non-metallic inclusions. Being of an entirely different nature from the metal they do not respond to anodic polishing, and the increase of current-density which takes place at the inclusion–metal boundaries results in a severe local attack. Even the smallest inclusions are affected in this way, but the attack varies with different electrolytes, some having the property of little attack on the inclusions; the colour of the inclusions is, however, invariably destroyed and replaced by black. For this reason steels appear somewhat less clean when electrolytically polished than when mechanically polished. Thus the results obtained from electrolytic polishing are not as good as those from mechanical polishing, when examining such specimens as wrought iron and free-cutting steels, etc. At this juncture it would be opportune to discuss the effect of electrolytic polishing on the graphite phase usually encountered in cast irons. During the course of this work many attempts were made to polish successfully specimens containing graphite in various electrolytes and at all ranges of current-density, but none was successful, the usual result being a mass of holes where the graphite phase had been attacked during electrolysis. The most promising electrolyte for this work was the perchloric acid–alcohol type suggested by De Sy and Haemers[9] when used at the maximum current-density possible (about 4 amp./sq. cm. with the equipment available), but even this was far from satisfactory. Cast irons containing no graphite, however, may be polished quite satisfactorily by electrolytic means.

Any cracks or discontinuities in the metal are also magnified somewhat by electrolytic polishing, and specimens being examined for such defects should always be polished mechanically. On the other hand, whilst electrolytic polishing may give an erroneous magnitude to such defects, it is possible that any fine cracks or films such as hair-line cracks or grain-boundary films, which may escape detection with ordinary mechanical polishing, may be easily revealed by an electrolytic polish. The amount of etching required by electrolytically polished specimens is substantially less than that required by mechanically polished specimens. Reference to Table I shows that the average thickness of metal removed in etching following electrolytic polishing was $0 \cdot 10\mu$, whereas the result for etching following mechanical polishing was $0 \cdot 25\mu$. Such results indicate that electrolytic polishing gives a nearer approach to the true structure of the metal than mechanical polishing. An illustration of this is shown in Fig. 23, which shows the very thin boundary films of large grains in an overheated 5% nickel case-hardening steel; such films are extremely difficult to reveal by mechanical polishing and etching.

Electrolytic polishing must of its very nature remove metal from the specimen surface in some atomic manner, and differs essentially from mechanical polishing in that no surface layer (amorphous or otherwise), such as that first described by Beilby,[10] occurs. In electrolytic polishing, the flowed or disturbed layer of metal produced by polishing on emery papers is not just thinned down as in the case of final mechanical polishing, but it is removed anodically in such a way that the resultant surface has better etching characteristics. With correct electrolytic polishing, repolishing, and etching are not necessary to obtain the true structure as is often the case with mechanical polishing. Electrolytic polishing is particularly useful for polishing alloys with single phases such as ferrite or austenite, which are usually fairly difficult to polish free of scratches by mechanical means. This is illustrated in Figs. 12 and 13, and also in Figs. 19–22 inclusive.

The attack by etching reagents on electrolytically polished specimens is much more active than on surfaces polished mechanically, more contrast being obtained between the structural constituents. This is illustrated in Figs. 6–13, which show familiar steel microstructures taken from specimens polished by both methods. Other familiar ferrous microstructures prepared by electrolytic polishing are shown in Figs. 14 and 15. These photomicrographs illustrate that specimens whose structure consists of more than one phase may be successfully polished electrolytically. Figures 16, 17, and 18 show the structure of a specimen of burnt mild steel polished in an electrolyte of the perchloric acid/alcohol type, and illustrate that electrolytic polishing gives good results when the specimen is examined at all magnifications. Figures 19 to 22 inclusive illustrate the microstructure of four types of steel sheets which were prepared together with others, in one mount. Although there were some twelve different types and tempers of steel sheet in this mount, all responded well to electrolytic polishing in an electrolyte of the 50% nitric acid/50% acetic anhydride type described later in this work. This example is given to illustrate the versatility of electrolytic polishing.

'Polish attack' has been mentioned earlier in

this work in connection with the effects of current-density, and temperature and age of the electrolyte. Figure 24 shows the characteristic but confusing relief effect, often of a fluted or wavy appearance, in the structure of a 0·3% carbon steel polished in Jacquet's solution. Figure 25 shows a good example of this defect in the unetched condition; etching of the specimen partially masks the defect but does not destroy it altogether. At present no logical explanation can be offered of this peculiar but interesting phenomenon which takes place independently of the type of structure or electrolyte. It is known, however, that it occurs when the current-density is a little too low for perfect polishing, when the temperature is higher than normal, or in the absence of agitation or in aged electrolytes. The defect can nearly always be prevented by increasing the current-density by approximately 25%.

Concluding this comparison of electrolytic polishing with mechanical polishing, the electrolytic method offers the rapid presentation without laborious and skilful effort, of microstructures entirely free from residual scratches and lack of polished finish which often characterize specimens polished mechanically. On the other hand, electrolytic polishing is not at the moment suitable for specimens containing graphite or large amounts of non-metallic inclusions, or cracks or discontinuities. Nevertheless, the two methods of polishing may be complementary to a large extent.

Search for Alternative Electrolytes

One object of this work was to investigate the potential value of electrolytes for polishing ferrous specimens other than those so far known to give satisfactory results. An extensive search of known and potential electrolytes was therefore undertaken. One type of specimen and cell only was used; the specimen consisted of mild steel in the normalized condition, all but the surface to be polished (area 3 sq. cm.) was vitreous-enamelled, and the cell used was that previously described and illustrated in Fig. 4. Agitation was used in every case, and in most cases the temperatures of the electrolytes were kept constant at 15°–20° C. to eliminate the effect of temperature on the viscosity and conductivity of the electrolyte and the resultant current-density required. The time employed was varied according to the current-density, so that the same amount of metal was removed anodically in each case. Values employed were as follows:

Current-Density, amp./sq. cm.	Polishing Time, min.
0·05	10
0·10	5
0·20	$2\frac{1}{4}$
0·50	1
1·00	$\frac{1}{2}$
2·00	$\frac{1}{4}$

The literature on electrolytic polishing reveals that the following acids have been suggested as forming the basis of electrolytes capable of polishing metals:

(1) Perchloric acid
(2) Nitric acid
(3) Chromic acid
(4) Phosphoric acid
(5) Sulphuric acid
(6) Citric acid, tartaric acid
(7) Hydrochloric acid, hydrofluoric acid

and that the following necessary dilutants have been suggested or used:

Acetic anhydride.
Acetic acid.
Alcohol.
Glycerine, dextrose, and other miscellaneous organic substances.

The water content of the electrolyte is also very important, and is usually between 5% and 20% at the time of preparation. With less than 5% of water the specific conductivity of the electrolyte is usually too low, and with more than 20% of water it is often difficult to obtain a range of current-density in which electrolytic polishing takes place at all.

(1) *Perchloric Acid Electrolytes*

Perchloric acid is one of the best electrolytic polishing acids and can be diluted with acetic anhydride, acetic acid, or alcohol. Dilution with acetic anhydride forms the basis of Jacquet's solution, which has many advantages including that of polishing over a fairly wide range of low current-densities, and also that of excellent covering power. Its disadvantages are its comparatively high cost, its explosive nature, and the long time and care needed in its preparation. Figures 7, 9, 11, 13, 14, and 15 show microstructures from specimens polished in this electrolyte.

Acetic acid may be used to dilute perchloric acid with advantage, little evolution of heat taking place. The solution is not quite as viscous as the acetic anhydride mixture but its covering power is almost equal and very good results can be obtained with it.

Alcohol can be used to dilute perchloric acid, and this type of electrolyte was evolved in Germany by De Sy and Haemers.[9] Practically no heat is evolved in mixing, which can therefore be done rapidly. This solution is not at all viscous and its covering power is very low; in operation it is preferable to have the specimen as near as possible to the cathode. The minimum current-density for anodic polishing is 2·0 amp./sq. cm., and consequently polishing is effected in 15 sec. The high polishing rate of this electrolyte enables a somewhat coarser preliminary finish to be employed, *e.g.*, grade 0 or 1F emery

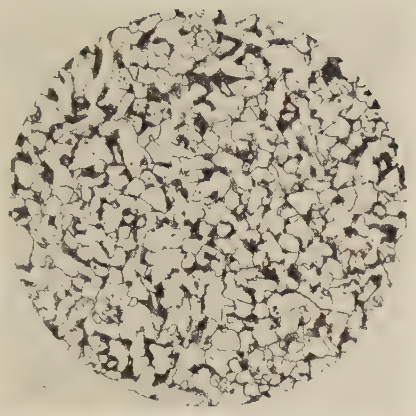

FIG. 6—Ferrite and pearlite in mild steel mechanically polished × 250

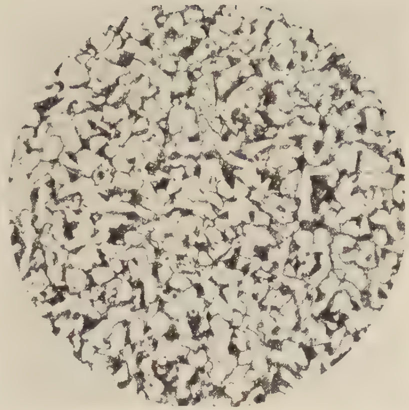

FIG. 7—The same steel as in Fig. 6 when electrolytically polished × 250

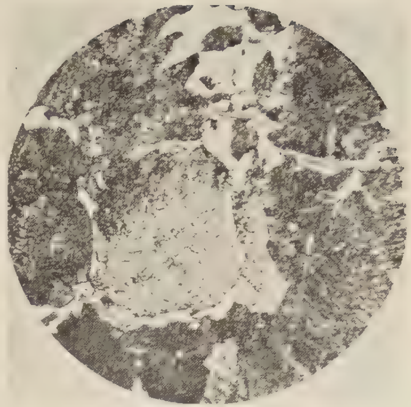

FIG. 8—Ferrite envelopes enclosing lamellar and sorbitic pearlite in annealed 0·50% carbon steel, mechanically polished, etched in 2% nital × 250

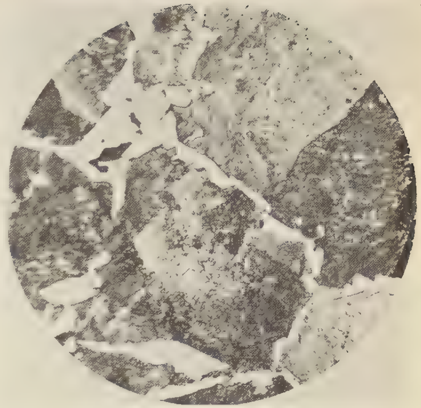

FIG. 9 The same steel as in Fig. 8 when electrolytically polished, etched in 2% nital × 250

Figures 6-13 reduced to three-quarters linear in reproduction

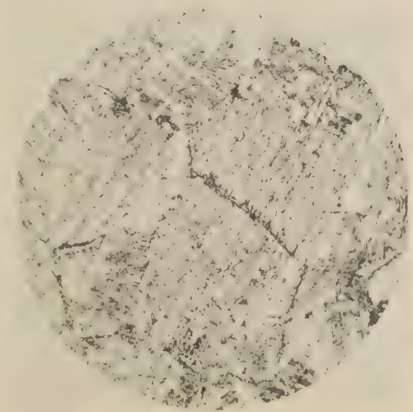

FIG. 10—Martensite with residual grain boundaries of austenite in 0·50% carbon steel, water-quenched; mechanically polished, etched in 2% nital × 250

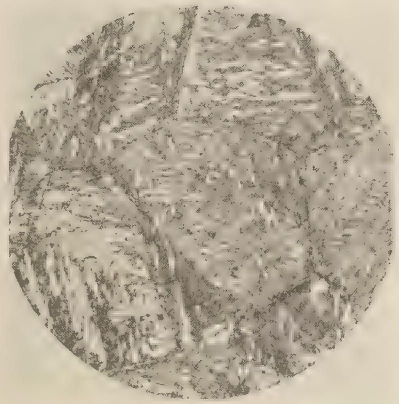

FIG. 11—The same steel as in Fig. 10 when electrolytically polished, etched in 2% nital × 250

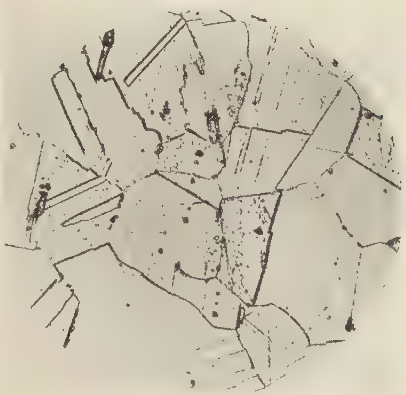

FIG. 12—Austenite in an 18% Cr/8% Ni stainless steel mechanically polished, etched in mixed acids in glycerine × 500

FIG. 13—The same steel as in Fig. 12 when electrolytically polished, etched in mixed acids in glycerine × 500

FIG. 14—Coarse lamellar pearlite with network of cementite in surface of case-hardened steel, untreated; electrolytically polished in Jacquet's solution, etched in 2 % nital × 500

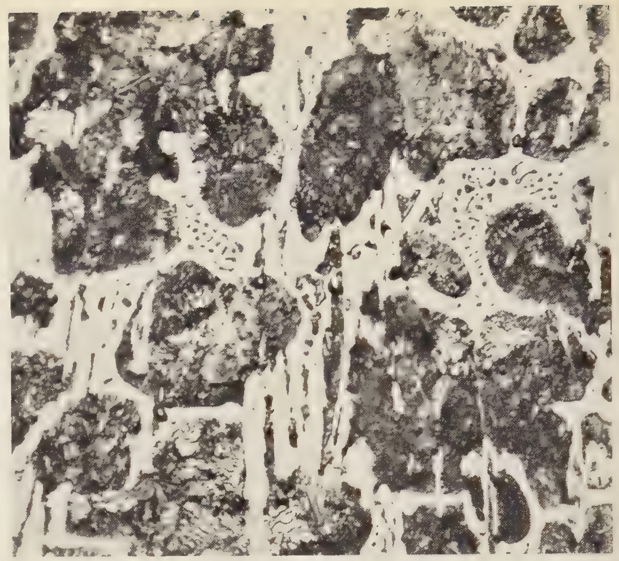

FIG. 15—Pearlite, cementite, and phosphide eutectic in structure of white cast iron electrolytically polished in Jacquet's solution, etched in 2 % nital × 500

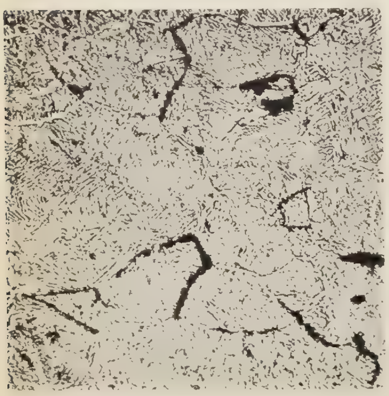

FIG. 16—Microstructure of burnt mild steel electrolytically polished in perchloric acid/alcohol solution, etched in 2 % nital × 25

FIG. 17—Microstructure of burnt mild steel electrolytically polished in perchloric acid/alcohol solution, etched in 2 % nital × 100

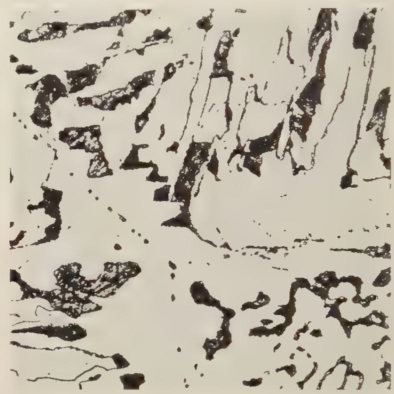

FIG. 18—The structure of burnt mild steel electrolytically polished in perchloric acid/alcohol solution, etched in 2 % nital × 500

Figures 16–18 reduced to four-fifths linear in reproduction

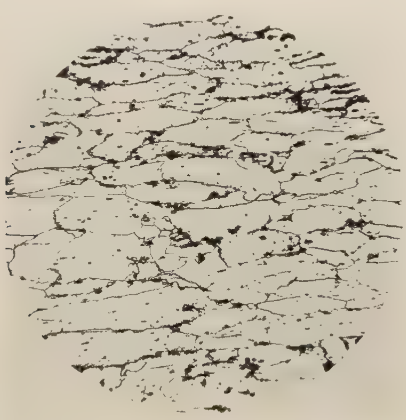

FIG. 19—Elongated ferrite and dispersed pearlite in 'hard-from-the-mill' steel sheet

FIG. 20—Equi-axed ferrite grains and divorced carbide in a normalized sheet of 'ingot iron' (carbon, 0·030 %)

Figures 19 and 20 illustrate the longitudinal microstructures of two types of steel sheet electrolytically polished in 50 % nitric acid/50 % acetic anhydride electrolyte for 15 sec. at 2 amp./sq. cm. current-density, etched in 2 % nital × 150

(Reduced to three-quarters linear in reproduction)

Merchant]

FIG. 21—Equi-axed ferrite and pearlite grains with small strings of inclusions in a low carbon killed-steel sheet

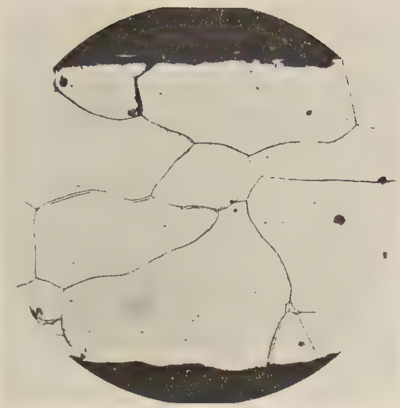

FIG. 22 Large silico-ferrite grains with some divorced carbide in a 4 %-silicon-steel transformer sheet

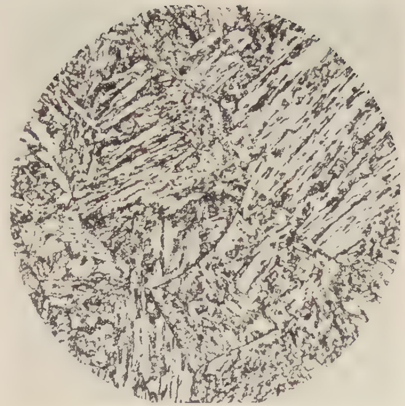

FIG. 23 5 %-nickel case-hardening steel in the normalized condition showing large grains occasioned by previous overheating; electrolytically polished, etched in 2 % nital × 250

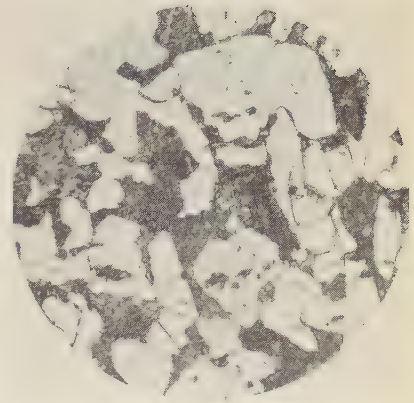

FIG. 24—0·3 %-carbon steel electrolytically polished exhibiting "polish attack" or waviness in the structure, etched in 2 % nital × 500

Figures 21 and 22 illustrate the longitudinal microstructure of two types of steel sheet electrolytically polished in 50 % nitric acid/50 % acetic anhydride electrolyte for 15 sec. at 2 amp. sq. cm. current-density, etched in 2 % nital × 150

FIG. 25—The same steel as in Fig. 24 when electrolytically polished but not etched, showing wavy or fluted structure associated with "polish attack" × 500

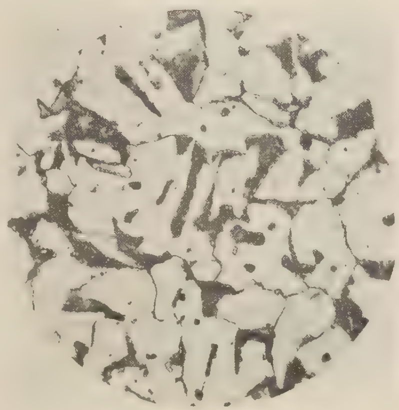

FIG. 26—Mild steel electrolytically polished in 10 % nitric acid/alcohol electrolyte, etched in 2 % nital × 500

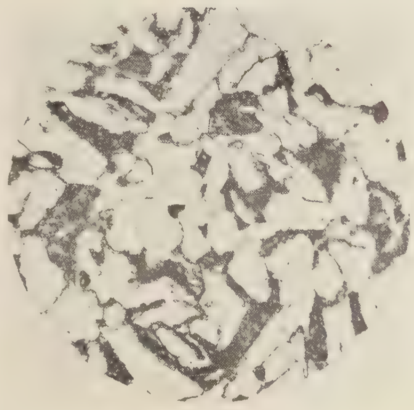

FIG. 27—Mild steel electrolytically polished in chromic acid/acetic anhydride electrolyte, etched in 2 % nital × 500

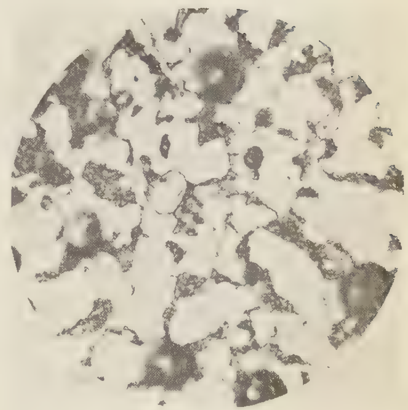

FIG. 28—Mild steel electrolytically polished in phosphoric acid/glycerine electrolyte, etched in 2 % nital × 500

Figs. 21-28 reduced to three-quarters linear in reproduction

paper finish. A further feature is that this electrolyte has less attack on inclusions and defects than the acetic anhydride or acetic acid types. It has the disadvantage of being fairly volatile and its composition soon changes unless care is taken.

In view of the explosive potentialities of perchloric acid, more attention was given to investigating electrolytes with other acids as the fundamental constituent ; nevertheless, it forms an electrolyte of the highest order for electrolytic polishing.

(2) *Nitric Acid Electrolytes*

Nitric acid was investigated as an electrolyte. Anodic polishing takes place in concentrated nitric acid at a minimum current-density of 1·5 amp./sq. cm., and fairly good metallographic results were obtained, but the concentrated acid (specific gravity 1·42 g./c.c.) is not a very desirable liquid to use as an electrolyte, and various dilutions with acetic anhydride, acetic acid, and alcohol were investigated.

Nitric acid should always be diluted with acetic anhydride by adding the latter carefully to the acid ; great heat is evolved, and the solutions should be cooled in mixing. The optimum or preferred solution was found to be 50% of nitric acid with 50% of acetic anhydride, which has a free water content of 9%. Excellent results were obtained at current-densities from 1·0 to 2·0 amp./sq. cm. ; Figs. 19 to 22 inclusive show photomicrographs of specimens of steel sheet polished in this electrolyte. The solution is fairly viscous but its relative covering power is poor.

Nitric acid may be mixed with acetic acid in any proportion without evolution of heat, and electrolytes with proportions of nitric acid from 20% to 80% (by volume) are capable of electrolytic polishing at current-densities from 1·0 amp./sq. cm. upwards. The optimum solution was one containing 40–50% of nitric acid ; this had a free water content of 14–17%. The results obtained from this electrolyte were not quite as good as those with nitric acid/acetic anhydride, but nevertheless, very satisfactory microstructures were obtained with it. One interesting feature of this electrolyte was that with current-densities less than those required for anodic polishing, the specimen became coated with an even and somewhat durable jet-black coating. On increasing the current-density a brightly polished annular ring appeared around a jet-black centre, thus illustrating the low covering power of the electrolyte, until eventually, with sufficient current-density, the specimen became polished all over.

Nitric acid may be diluted with alcohol by adding the former to the latter, and mixtures containing up to 33% of nitric acid were tried out at various current-densities. In this work colourless 'industrial spirit' was used for alcohol and it was found that 5% of ether added to this before mixing with the acid appeared to improve the results obtained. The optimum solution was that containing 10% of nitric acid, and this gave excellent results at current-densities exceeding 1·5 amp./sq. cm. ; a photomicrograph of a specimen of mild steel polished in this electrolyte is shown in Fig. 26. Polishing takes place in 15–20 sec., thus enabling somewhat coarser preliminary finishes to be employed, and specimens may be progressed from the ground state to the microscope in less than 2 min. Owing to the short electrolysis time and the low viscosity of these solutions, it was observed that agitation was not quite so vital as in the case of other electrolytes. This electrolyte, however, like its perchloric counterpart, has a poor covering power and high volatility.

It was observed that 5% of nitric acid in alcohol, which has been used by the metallographer of iron and steel as an etchant, forms quite a good electrolyte for polishing purposes, but it was very difficult to prevent etching of the specimen, even though it was removed from the electrolyte with the current still on and washed immediately. Specimens were observed to be etched very well indeed but somewhat deeper than usual ; it is considered that some etching takes place during the course of electrolysis. With higher concentrations of nitric acid, even 10%, the tendency to etch during electrolysis was suppressed.

Nitric acid is of great potential value as an electropolishing acid, and results as good as those from Jacquet's solution are obtainable from its dilutions with acetic anhydride and alcohol. In addition nitric acid is cheaper and less dangerous to handle than perchloric acid, and electrolytes of which it forms the base give more freedom from polish attack and attack on the inclusions. With all nitric acid electrolytes, increasing the nitric acid content increases the free water content and therefore the conductivity, and thus reduces the voltage required to give the optimum current-density, but increases the current-density at which anodic polishing takes place. All nitric acid electrolytes recommended above offer rapid polishing of specimens at relatively high current-densities.

(3) *Chromic Acid Electrolytes*

Chromic acid (a 75% solution of chromic trioxide in water) showed slight signs of anodic

polishing when used alone, and as it is a powerful oxidizing acid its dilution with acetic anhydride and acetic acid was investigated. It may be diluted with acetic anhydride by adding the latter carefully, a drop at a time, and cooling during mixing. Various combinations were investigated, but the preferred solution was found to be 20% of chromic acid with 70% of acetic anhydride and 10% of added water, and with this solution anodic brightening took place at current-densities varying from 0·10 to 2·0 amp./sq. cm. The solution is fairly viscous and its covering power is fairly good. A specimen of mild steel polished in this electrolyte is shown in Fig. 27.

Chromic acid may be diluted with acetic acid without evolution of heat, and with a solution containing 20% of chromic acid and 80% of acetic acid, quite good electropolishing results were obtained at current-densities ranging from 0·50 to 2·0 amp./sq. cm. This solution has a higher free water content than its acetic anhydride counterpart, however, and its covering power is not as good.

Chromic acid cannot be diluted with alcohol but its dilution with acetic anhydride or acetic acid shows that it must be recognised as of some potential value as an electrolytic polishing acid.

(4) *Phosphoric Acid Electrolytes*

Phosphoric acid (ortho-phosphoric acid, H_3PO_4) has been mentioned frequently in the literature for the electrolytic polishing of copper,[11] iron or silicon-iron specimens containing a single phase,[6] and stainless steels.[12] In this work, the acid was tried alone and when diluted with glycerine, alcohol, acetic anhydride, acetic acid, and sulphuric acid.

When used alone at both room and higher temperatures anodic brightening takes place at current-densities of 0·10 amp./sq. cm. and upwards. The optimum current-density is about 0·75 amp./sq. cm., and above this value a troublesome pitting or polish attack takes place. This polish attack occurs, not as the 'wavy' type previously mentioned, but as a characteristic 'spotty' appearance under the microscope, the spots, or holes as they actually are, being somewhat controlled by the current-density and polishing time. A typical example of this is shown in Fig. 28. It was not possible to avoid this defect with any phosphoric acid or sulphuric acid electrolyte.

The use of phosphoric acid and glycerine has been suggested by Uhlig,[13] and an investigation of various mixtures showed that the preferred solution was 80% of phosphoric acid with 20% of glycerine. This had a free water content of approximately 6%, and was very viscous indeed and had an excellent covering power. Optimum polishing results were obtained at a current-density of 0·50 amp./sq. cm., and a specimen of mild steel polished in this electrolyte is shown in Fig. 28, which, it may be observed, is not without the spotty polish attack mentioned above.

Very little success was obtained by diluting phosphoric acid with alcohol. A mixture of 20% of phosphoric acid and 80% of alcohol showed signs of anodic brightening but had a very low conductivity indeed, which was not improved by additions of water up to 20%. Residual scratches and spotty polish attack were always present. Dilution of the acid with various proportions of acetic anhydride and acetic acid likewise proved unsuccessful; the solutions had a very poor conductivity, even with an added water content, and no anodic brightening took place; instead, a thick brown film formed on the specimen which became deeply etched and pitted.

Of many electrolytes based on phosphoric acid which were tried, the most satisfactory was its dilution with glycerine, but even here the current-density to produce even a minimum of the spotty polish attack was very critical. Although high temperatures have been advocated for the use of phosphoric acid electrolytes, it was found that equal results were obtained from its use at room temperatures, with the added benefits of temperature and current-density control. One interesting point about phosphoric acid electrolytes was the long time and large amount of metal to be removed anodically from the surface to erase the scratches. Theoretically, and according to experience with other electrolytes, polishing should be effected at a current-density of 0·50 amp./sq. cm. in one min.; yet with the phosphoric acid/glycerine electrolyte, all scratches were not removed until the specimen had been polished for 10–15 min. This feature of phosphoric acid electrolytes is a distinct disadvantage.

(5) *Sulphuric Acid Electrolytes*

The use of sulphuric acid for electrolytic polishing, especially for commercial finishes has been mentioned frequently in the literature.[14, 15] Its use for metallographic polishing purposes was investigated when diluted with water, glycerine, acetic anhydride, acetic acid, phosphoric acid, and chromic acid. When diluted with various percentages of water and also glycerine, only very slight signs of anodic brightening were obtained at both room and elevated temperatures, the specimen becoming etched and pitted in most cases.

Sulphuric acid may be mixed with acetic anhydride with care; some heat is evolved, and the resulting solution is quite viscous and has a

fairly good covering power. The optimum solution was found to be 20% of sulphuric acid with 60% of acetic anhydride and 20% of added water, and fairly good results were obtained with it when used at current-densities varying from 0·20 to 0·50 amp./sq. cm., but the polishing time was much longer than that necessary with perchloric or nitric acid/acetic anhydride solutions. Similar results were obtained from a solution of 20% of sulphuric acid with 70% of acetic acid and 10% of water, but both electrolytes do not compare favourably in operation or results obtained with their perchloric or nitric acid counterparts.

Mixtures of sulphuric acid with phosphoric acid have also been widely suggested in the literature for the commercial polishing of stainless steels,[16, 17, 18] and various mixtures of these acids with water contents of 10–20% were investigated. These solutions were very viscous and had a good covering power, but their conductivity is high compared to other types of more successful electrolytes. Anodic brightening took place over a wide range of current-densities (from 0·20 to 2·0 amp./sq. cm.), but in all cases spotty polish attack occurred, and long polishing times were required. Electrolytes containing less than 50% of phosphoric acid did not respond at all well, and at normal temperatures and low current-densities, these sulphuric-rich mixtures gave rise to high polarization, unstable conditions, and the formation on the specimen of a hard yellow film. With the same current-densities at higher temperatures, the film did not form but the specimen became etched and pitted. Additions of 5% of glycerine to the more promising sulphuric–phosphoric mixtures showed slightly improved results but the polishing time increased unfavourably. Five per cent. of chromic acid was also added to the most promising mixtures, and whilst this addition resulted in slightly brighter results, it was not possible to avoid spotty polish attack at any combination of current-density and time.

In general, the most promising electrolytes based on sulphuric acid were those of its dilution with acetic anhydride or acetic acid, but even these did not compare with perchloric or nitric acid base electrolytes, and the polishing time was increased to more than twice normal. The various mixtures of sulphuric acid and phosphoric acid, although widely proposed in the literature for commercial polishing, did not give results satisfactory for metallographic examination.

(6) *Citric and Tartaric Electrolytes*

The use of these "acids" as possible solutions for electrolytic polishing was investigated because citric acid has been mentioned as a definite constituent of a successful electrolyte for the commercial polishing of stainless steel.[19]

Citric acid alone and also when diluted with alcohol and glycerine gave negative results. Dilution with 20% of phosphoric acid also gave little success. When diluted with 20% of sulphuric acid fairly good signs of anodic brightening were observed at current-densities varying from 0·20 to 2·0 amp./sq. cm. When diluted with acetic anhydride and water, the resulting solution was very poorly conductive and polishing did not take place at the maximum current-density available (0·20 amp./sq. cm.). A solution of 20% of citric acid and 20% of sulphuric acid with 50% of acetic anhydride and 10% water, mixed with care, gave fairly satisfactory anodic brightening when used with a current-density from 0·20 to 2·0 amp./sq. cm., but the polishing time required was longer than normal and some signs of spotty polish attack were evident.

Tartaric acid was also investigated in the same way as citric acid, but even poorer results were obtained; in addition, tartaric acid cannot be mixed cold with 20% of sulphuric acid without precipitation occurring. It was not expected that these weak organic "acids" would prove useful by themselves as bases for potential electrolytes, but citric acid certainly appeared useful as an addition agent to sulphuric acid for such purposes. Such electrolytes, however, are not suitable for metallographic polishing purposes.

(7) *Hydrochloric and Hydrofluoric Acid Electrolytes*

Hydrochloric acid was investigated alone and when diluted with various proportions of alcohol, glycerine, and acetic anhydride; some signs of anodic brightening and scratch removal were obtained but generally the results were very poor when compared to other electropolishing acids. One difficulty may be that of the high water content of the laboratory acid, and in this respect higher proportions of the dilutants gave better results. Although hydrochloric acid is not an oxidizing acid, the possibility of its use as a potential electrolyte for electropolishing should not be overlooked.

Hydrofluoric acid was investigated alone and when diluted with alcohol, glycerine, and sulphuric acid. Both alone and with alcohol, no signs of polishing were apparent; dilution with 60% of glycerine, however, showed some signs of anodic brightening. A 10% solution of hydrofluoric acid with 40% of sulphuric acid, the remainder being water, also gave signs of anodic brightening, but this solution had a very high conductivity which is generally a sign of poor performance as far as metallographic polishing is concerned.

Hydrofluoric acid cannot be diluted with acetic anhydride, but a dilution with 80% of acetic acid gave fairly bright results, although accompanied by some polish attack. Generally, although it shows signs of being a potential electrolyte for polishing steel, hydrofluoric acid is similar to hydrochloric acid in not approaching the results obtained with electrolytes based on perchloric acid, nitric acid, etc., and is in any case difficult and obnoxious to handle.

Recommended Electrolytes

On the results of this work a list of recommended electrolytes together with their properties, working conditions, and relative metallographic response was prepared, and is shown in Table II. Specific gravity measurements are a useful guide to the age of the electrolyte. The relative viscosity and covering power shown are from visual observations only, but appear to be well connected. The free water content is considered important, and is based on the following approximate water content of the chemicals used :

60% perchloric acid ...	40%
Conc. nitric acid ...	32%
Chromic acid	25%
Acetic anhydride ...	5%
Acetic acid	2%
Alcohol	5%,

and the fact that acetic anhydride takes up water to form acetic acid when used to dilute such oxidizing acids. The relative cost per litre of the electrolytes is interesting in that Jacquet's solution is the most expensive ; all the nitric acid electrolytes, on the other hand, are fairly cheap. Acetic anhydride electrolytes are difficult to prepare ; acetic acid electrolytes are easier to prepare and much cheaper, but perhaps not quite as good ; alcohol electrolytes are cheap and easy to prepare, but are volatile and have poor viscosity and covering power, and require fairly high working current-densities.

Conclusion

In the course of this work certain conclusions became apparent which might bear on the theory of electrolytic polishing which is by no means yet fully understood. In electrolytic polishing, the anodic dissolution has a levelling-out effect on the roughness of the surface, which is in marked contrast to the usual course of the solution of metals where etching and corrosion and a roughening of the surface are the characteristic phenomena. The present theory of electrolytic polishing involves the selective removal of elevated portions of the surface, but the fact that a specimen, which has already been perfectly polished mechanically or electrolytically, may be repolished electrolytically without detriment to its surface, seems to indicate that some modification of this theory is required. Electrolytic

Table II—*Recommended Electrolytes*

Electrolyte	Preparation	Specific Gravity, g./c.c.	Relative Viscosity	Covering Power	Free Water Content, %	Prime Cost per Litre, shillings	Optimum Working Current-Density, amp./sq. cm. Minimum	Maximum	Relative Metallographic Response
Perchloric acid/acetic anhydride (Jacquet's solution)	765 c.c. acetic anhydride added carefully to 180 c.c. of 60% perchloric acid, followed by 55 c.c. of water	1·165	Quite viscous	Excellent ...	3	15	0·05 for 10 min.	0·20 for 2·5 min.	Very good indeed
Perchloric acid/acetic acid	800 c.c. of glacial acetic acid added to 200 c.c. of 60% perchloric acid	1·170	Fairly viscous	Very good...	10	11	0·20 for 2·5 min.	0·50 for 1·0 min.	Very good
Perchloric acid/alcohol (Originated by De Sy and Haemers)	60 c.c. of 60% perchloric acid and 140 c.c. of water added to 800 c.c. of alcohol containing 3% of ether	0·920	Poor	Poor ...	20	3	2·0 for 15 sec.	...	Very good indeed
Nitric acid/alcohol ...	100 c.c. of conc. nitric acid added to 900 c.c. of alcohol containing 5% of ether	0·880	Poor	Poor ...	7	1	1·5 for 20 sec.	2·0 for 15 sec.	Excellent
Nitric acid/acetic anhydride	500 c.c. of acetic anhydride added carefully to 500 c.c. of conc. nitric acid	1·225	Fairly viscous	Fairly poor		6	1·0 for 30 sec.	2·0 for 15 sec.	Excellent
Nitric acid/acetic acid	500 c.c. of glacial acetic acid added to 500 c.c. of conc nitric acid	1·220	Fairly viscous	Fairly poor	17	3	1·5 for 20 sec.	3·0 for 10 sec.	Very good
Chromic acid/acetic anhydride	700 c.c. of acetic anhydride added carefully to 200 c.c. of 75% chromic acid followed by 100 c.c. of water	1·150	Fairly viscous	Fairly good	6	8	0·20 for 2·5 min.	2·0 for 15 sec.	Very good

polishing can proceed, using a suitable clean specimen and ideal polishing conditions, well beyond the stage at which all scratches are erased, and yet it is not possible to distinguish the structural constituents of the metal without some etching operation. If the conditions are not ideal for electrolytic polishing, however, scratch removal may occur, and the constituents of the metal may well be revealed ; this occurs in certain electrolytes of low acid content, and also with satisfactory electrolytes if a higher current-density than normal is used. In ideal electrolytic polishing, the fact that the constituents are not revealed seems to suggest that a modified surface layer of metal is present, similar to but much thinner than that which occurs in even 'perfect' mechanical polishing. It is the nature of this modified surface layer produced by electrolytic polishing which is not yet fully understood.

Voltage and Current-Density

In successful electrolytes for polishing ferrous specimens, the voltage across the cell is unimportant and is only used to control the current-density. The latter controls the anodic film necessary for polishing, and it appears that a minimum concentration of ions is necessary for film formation. At a current-density lower than that required for polishing, etching takes place ; it is possible that all the anodic products are highly soluble in the electrolyte with such conditions. At current-densities slightly lower than that required for optimum polishing, the curious wavy or fluted structure sometimes appears, but as yet there is no explanation for this phenomenon. At current-densities too high for optimum polishing, gas evolution and pitting occur ; under such conditions the anodic film is probably continually being destroyed. High current-densities will also cause local overheating of the anode and destroy polishing conditions.

Bath Conditions

The temperature, agitation, and age of the electrolyte are only important in so far as maintaining the correct current-density for polishing. Increased temperature increases the current-density required, and decreases the viscosity of the electrolyte. Agitation assists in maintaining uniform conditions at the anode. In use, the acid radicles of the electrolyte are used up and the electrolyte becomes charged with iron radicles, and so current-density conditions for optimum polishing are changed.

Surface Preparation and Time

The degree of surface preparation controls the polishing time required, which may also be varied with the current-density employed. Some electrolytes such as Jacquet's solution, operate at low current-densities, which entail a longer polishing time ; a fine degree of surface preparation is, therefore, useful in keeping this at a minimum. Other electrolytes such as those based on nitric acid, work only at relatively high current-densities ; because polishing is very rapid, a relatively coarsely prepared surface may suffice. Some electrolytes, *e.g.* those based on phosphoric or sulphuric acid, operate so relatively slowly that coarse surface irregularities are not removed ; these are not suitable for metallographic polishing purposes.

Specimen Composition

A wide range of ferrous specimens may be polished electrolytically, the exceptions being those containing graphite or large amounts of non-metallic inclusions or cracks or discontinuities. Some electrolytes, however, especially those based on nitric acid, have the property of minimum attack on the inclusions. Any size of specimen may be polished providing the conditions are correctly obtained.

Electrolyte Composition

An ideal electrolyte for metallographic polishing should be such that polishing takes place in a reasonable time ; it should be capable of operating at normal temperatures and should remain constant in composition and have a reasonable life. It should be cheap and easy to prepare, and should not be obnoxious or dangerous in use. Its conductivity is also important, and should be such that the required current-density is obtained with a reasonable voltage.

For successful metallographic polishing, the electrolyte must have in its composition three essentials which are (1) an acid, preferably of an oxidizing character ; (2) a solvent in which the anodic products are soluble, and which also acts as a dilution agent ; and (3) a limited and definite amount of water. Of the acids investigated in this work, perchloric acid, nitric acid and chromic acid form the best electrolytes for metallographic polishing. Phosphoric acid and sulphuric acid are not suitable for this purpose owing to their tendency to produce pitting or spotty polish attack ; in addition both acids are relatively slow in polishing and they do not mix well with dilution agents. The right kind of acid ion such as perchlorate, nitrate, or chromate is, therefore, the first essential for successful polishing. The second essential constituent is a liquid in which the anodic products are soluble, and in this respect acetic anhydride, acetic acid, and alcohol may be classed as active solvents of oxidized compounds ; the most successful electrolytes investigated in this work contained one of

these dilution agents. Acetic anhydride and acetic acid are viscous in character, which condition favours anodic film formation, and also covering power ; alcohol, on the other hand, is not at all viscous. Acetic anhydride gives greater control over the free water content of the electrolyte than either acetic acid or alcohol, inasmuch as the anhydride takes up water, forming acetic acid when added to respective acids. In this way, each 100 c.c. of acetic anhydride used takes up approximately 18 c.c. of water from the electropolishing acid, although it may donate about 5 c.c. of water as impurity in the anhydride. When using acetic acid or alcohol as dilution agents, the free water content of the electrolyte is mostly dependent on the water content of the acids used. Electrolytes with lower free water contents are thus possible by using acetic anhydride. All these dilution agents probably change the anodic film resistivity and at the same time give added importance to the free water content, the ions of which together with those of the acid, are the current carriers of the cell. The current-density for optimum polishing increases with the free water content, which in turn decreases the time required for polishing ; nevertheless, too high a free water content may destroy polishing conditions altogether and only an etching action will result.

The Anodic Film

When ferrous specimens are polished in an electrolyte containing perchloric acid and acetic anhydride or acetic acid, a transparent viscous brown film appears on the specimen. No such marked film was apparent with other successful electrolytes mentioned in this work, *e.g.* perchloric acid/alcohol or the nitric acid electrolytes, but it is significant that all these electrolytes operate at a much higher current-density, and it is probable that an anodic film does exist with such electrolytes. These anodic films may have a colloidal composition and possibly form by the oxidation of ferrous ions by the acid ions at the anode, combining also with the dilution agent and the water present. The factors controlling these films are current-density, specimen composition, and temperature and viscosity of the electrolyte. The last named has a bearing on the covering power of the electrolyte, and electrolytes of poor covering power require high current-densities for polishing, whilst those of good covering power work well at low current-densities. The specimen composition exerts an influence on the anodic film inasmuch that if there are present in the metal two widely different phases, such as ferrite and graphite, the continuity of the film may be destroyed and polishing will not be effected. A good deal of research into the physical chemistry of anodic film formation as it affects electrolytic polishing is indicated.

There is little doubt that electrolytic polishing, although it is perhaps not yet fully understood, represents a marked advance in preparing polished surfaces, especially as its application is not confined to metallography but also concerns other branches of science.

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Some Experiments on Corrosion of Steel by Boiling Water*

By A. J. Gould, Ph.D.,† *and U. R. Evans, Sc.D., M.A.*†

SYNOPSIS

The behaviour of steel in boiling water is shown to be determined largely by the position of the formation of solid corrosion products. Under anaerobic conditions steel reacts at first with water, eliminating hydrogen, but a film (first probably ferrous hydroxide, then becoming magnetite) is formed over the surface, so that the attack over long periods is very slight. In the presence of oxygen the conversion of ferrous hydroxide to magnetite, or rust, appears to take place to some extent at a distance from the metal, so that the film is discontinuous and serious corrosion occurs. Salts tend to increase, and sodium hydroxide to diminish, the attack under aerobic (but not under anaerobic) conditions. Copper deposited on part of the steel increases the intensity of attack in short experiments but not in long ones. Oxide films formed by previously heating in air, if interrupted by a scratch-line, can greatly increase the intensity of the attack.

INTRODUCTION

THE corrosion of steel by boiling water in presence of oxygen is generally assumed to conform to the mechanism established for attack by cold water. It is difficult, however, to explain why traces of oxygen in cold water produce general corrosion, whereas in boiling water they cause pitting. The difficulty partly disappears if it is accepted that the primary effects of boiling water on steel are the liberation of hydrogen and the production of an oxide film on the metal; for this assumption the work of Thiel and Luckmann [1] provides much support. The well-known action of oxygen in stimulating attack (which Denman and Bartow [2] found to be proportional to the oxygen concentration) may then be ascribed to the conversion of dissolved ferrous hydroxide to magnetite, or rust, at a slight distance from the surface, preventing the formation of a continuous film, which under anaerobic conditions would stifle attack.

To test this view the experiments described have been carried out. It was also sought to discover why magnesium salts, which retard corrosion at low temperatures as shown by Friend, Brown, and Barnet,[3] and by Evans,[4] sometimes stimulate attack at the boiling point, as emphasized by Ost,[5] by Bauer,[6] and by Bengough; [7] also why copper salts, which often exist in boiler feed water without apparently causing trouble, sometimes seem to be associated with intense attack.

MATERIALS

The steel *US*2 was prepared for another research[8] by the late Dr. T. Swinden; it was 0·3 mm. thick and had been annealed after rolling. Analysis showed :

C, %	Mn, %	Si, %	S, %	P, %
0·26	0·36	0·207	0·031	0·038

Specimens were ground with emery paper (Oakey No. 1) and washed several times in acetone. The distilled water was condensed in glass, from an electrically heated still. In the anaerobic experiments it was reboiled with 'bumping' just before use.

METHOD

Since the object was to study the mechanism of corrosion, no attempt was made to imitate boiler conditions; the geometry was kept simple, partially immersed specimens being used in view of the special phenomena often met with at the water-line. Weighed steel strips measuring 7·5 cm. × 1·0 cm. were placed vertically in glass boiling tubes of diameter 2·5 cm. and height 15·0 cm., the lower part of the tubes being immersed in a bath of boiling water (Fig. 1). The steel was immersed to a depth of 4·0 cm. in the water or solution to be tested, the upper part being surrounded by steam in the anaerobic experiments, but by a mixture of air and steam in the aerobic experiments. The pressure was about 1 atm., and the temperature of the boiling liquid varied from 96° to 100° C. in different experiments. It may be possible later to carry out experiments at higher pressures, and if so, it is hoped these will be conducted with specially purified water and with special attention to various

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† Cambridge University.

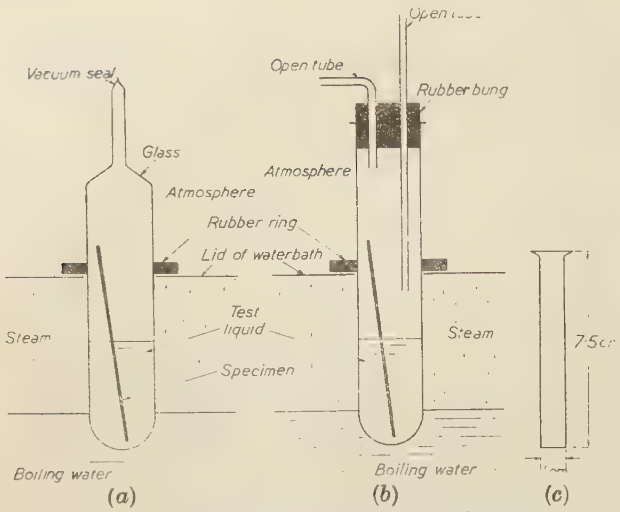

Fig. 1—Apparatus for studying the behaviour of steel towards boiling water ; (a) under anaerobic conditions, and (b) under aerobic conditions. (c) Detail of test specimen

factors which the present research showed to be important.

In starting an anaerobic experiment, the tube containing cold water or solution (previously boiled to expel oxygen) was evacuated with a ' Hivac ' pump ; when a leak tester indicated that the vacuum was satisfactory, the exit tube was sealed in a blowpipe flame. When terminating an anaerobic experiment, the whole boiling tube was immersed in boiling distilled water (previously boiled with ' bumping ') and the glass seal was broken. The evacuated space soon became filled by the entering water, leaving a bubble at the top, the volume of which was subsequently measured at atmospheric temperature and pressure. In many cases it was shown to consist largely of hydrogen, but impurities (recognizable by their odour) were present, and sometimes some air which had leaked in or escaped elimination. Occasionally violent explosions occurred when the hydrogen was mixed with excess of oxygen and heated, preventing measurements from being obtained.

The rubber bungs used in the aerobic experiments had received a prolonged treatment in warm dilute sodium hydroxide to remove sulphur from the surface layers, and during the tests air was sucked through each corrosion cell for 1 min. every day.

Table I—*Results of Anaerobic Exposure of Steel* US2 *to various Corroding Media*

Test	Corroding Medium	Duration of Test, days	Weight Change after Corrosion, mg.				Weight Change after Pickling, mg.		
			Sample				Sample		
			1	2	3	4	1	2	3
I	Distilled water	1	+0·4	+0·1	−0·1	...	...	...	...
		2	+1·0	+0·4	+0·6	...	...	...	...
		4	+1·7	+0·4	+0·1	+1·1	...	...	...
		6	...	...	...	...	−4·4	−3·0	−0·5
		8	+0·6	+0·8	...	...	...	...	...
		16	+2·5	+4·1	+2·6	...	...	...	...
		76	+6·4	+6·4	...	...	−0·6	−0·3	...
II	M/100 sodium hydroxide	4	+0·5	...	...	...	...	...	...
		6	+1·5	+1·5	...	...	−3·4	−2·6	...
		76	+5·6	+5·8	...	...	+1·5	−2·4	...
III	M/100 sodium chloride	4	+2·8	...	...	...	...	...	...
		16	+2·2	+4·5	...	...	...	...	...
		76	+7·0	+6·8	...	...	+1·5	+0·2	...
IV	M/100 sodium sulphate	4	>+1·4	+2·0	+0·2	...	...	...	...
		6	+1·7	−6·4	...	...	−4·2	−13·3	...
V	M/100 magnesium sulphate	4	+2·2	+1·8	+0·8	...	...	...	...
		76	+1·8	+2·8	...	...	−4·6	−5·9	...
			Half-coppered Specimens						
VI	Distilled water	6	+2·4	+1·0	...	...	−21·7	−18·5	...
		76	+6·6	+5·0	...	...	+0·6	−2·2	...
VII	M/100 sodium sulphate	6	−6·8	+1·4	...	...	−30·7	−15·8	...

TABLE II—*Results of Aerobic Exposure of Steel US2 to various Corroding Media*

Test	Corroding Medium	Duration of Test, days	Weight Change after Corrosion, mg.			Weight Change after Pickling, mg.			
			Sample			Sample			
			1	2	3	1	2	3	4
VIII	Distilled water	1	+1·4	...	...	...	...	...	...
		4	+5·4	+5·4	+3·6	...	...	...	...
		6	+6·6	+7·0	...	−11·7	−15·3	−29·0	−37·0
		76	+228·0*	...	...	−365·2	...	...	...
IX	$M/10$ sodium hydroxide ...	4	+1·7	+1·0	...	...	...	...	...
X	$M/100$ sodium hydroxide ...	6	+2·2	+3·4	...	−8·2	−8·7	...	...
		76	+78·5*	...	...	−119·9	...	...	...
XI	$M/10$ sodium chloride ...	0·25	+0·8	+1·0	...	...	...	...	...
		1	+2·5	...	...	...	...	...	...
XII	$M/100$ sodium chloride ...	16	+44·8	+188·6	...	...	...	...	...
		76	+463·6*	...	...	−686·5	...	...	...
XIII	$M/100$ sodium sulphate ...	6	+1·9	+8·5	...	−84·9	−71·2	−79·3	−104·0
XIV	$M/100$ magnesium sulphate	76	+354·4*	...	...	−648·9	...	...	...
			Half-coppered Specimens						
XV	Distilled water	6	+3·9	+5·9	...	−42·9	−47·3	...	...
		76	+250·2*	+249·3*	...	−351·7	−331·8	...	...
XVI	$M/100$ sodium sulphate ...	6	+33·0	−1·9	...	−99·8	−93·6	...	...

* Including sediment

Most of the experiments were carried out in duplicate. In some liquids a series of experiments were carried out terminating after different periods, *e.g.*, 1, 2, 4, 8, and 16 days; a few experiments were continued for 76 days. The gain in weight was always measured, and sometimes the specimens were afterwards cleaned by pickling and reweighed to obtain the loss in weight. In other cases, the film left on the specimen was transferred to celluloid in a cell similar to that already described by Evans,[9] but modified to dispense with the diaphragm.

About 170 specimens were tested. The measurements are recorded in Tables I and II,* but the conclusions which are summarized later were based mainly on qualitative observations, and particularly on the place where the corrosion product was thrown down, since as already explained, it is the site of precipitation which makes all the difference between stifling and continued attack. The weight changes, however, possess some interest, particularly the marked contrast between the figures obtained in the 76-day tests under anaerobic and aerobic conditions respectively. The poor agreement sometimes met with between duplicate experiments is not surprising, since the number of 'events' (*i.e.*, breakdowns) was small, and as explained elsewhere,[10] considerable scatter must then be expected. In addition, however, to random errors, deposition of silica in the long-period experiments probably made the weight gains too large and the losses too small.

* The weight changes in the two-stage experiments, although determined, are not included in Tables I and II, as the main object of these experiments was to observe whether intensification occurred at the scratch-line.

ANAEROBIC EXPERIMENTS

Distilled water produced on the steel a visible film, thicker below the water-line than above it and often exhibited interference tints; the liquid remained clear, and no rust or magnetite granules appeared. After a few days spots appeared on the steel, suggesting local breakdown of the film, and tiny particles of tenuous film which had probably peeled from the metal appeared in the liquid. Gas was evolved in the early stages in somewhat

variable amounts, but after 76 days the volume was about 1 ml. in a boiling tube of 40 ml. capacity, where the volume of the solution was about 13 ml. The specimens gained in weight, and the absence of any serious amount of loose product, even after 76 days, proved that the film formed on the surface had become extremely protective. Transfer of this film to celluloid showed it to be largely magnetite; it was strongly magnetic and contained both ferrous and ferric iron. The thinner film, formed above the water-line, was ferric hydroxide, presumably owing to a trace of oxygen in the steam.

Specimens exposed to boiling *M*/100 sodium hydroxide preserved their brightness better than those in water, and gave fewer flakes of stripped film. A comparison of the loss of weight after pickling, with the corresponding figures obtained with distilled water does not suggest that alkali greatly reduced attack under anaerobic conditions.

Experiments in *M*/100 sodium chloride, *M*/100 sodium sulphate, or *M*/100 magnesium sulphate also produced no rust and no rapid formation of loose corrosion product, provided that air was absent. A dark film appeared in sodium or magnesium sulphate, but the changes in weight were small. Specimens exposed for 76 days and then pickled, showed a small loss of weight in the case of magnesium sulphate. In sodium chloride or hydroxide there was sometimes a slight gain in weight due to a small amount of an adherent white deposit (probably silica) at the water-line.

Thus, in absence of oxygen, boiling water produces no serious attack on steel, alkali confers little benefit, and salts cause little harm. The rapid stifling of attack is understood if it is agreed that the water acts on the iron to produce ferrous hydroxide and hydrogen. When the liquid has become saturated with ferrous hydroxide, any further attack may be expected to produce a solid film over the surface; this is probably ferrous hydroxide at the outset, which later decomposes to produce hydrogen and magnetite thus:

$$3\ Fe(OH)_2 = Fe_3O_4 + 2H_2O + H_2\ ;$$

the magnetite, formed *in situ*, still constitutes a protective film shielding the metal from attack. In the early stages the film peels off locally, but doubtless fresh film can always be formed at the spots thus exposed, and after a time (probably when the stresses left by the grinding have become exhausted) the peeling seems to cease.

Aerobic Experiments

Very different were the results when air was present. Distilled water soon gave large quantities of granular magnetite and flocculent rust; in some special experiments where glass-wool wads had been placed at the water-line, the magnetite was found partly within the glass wool, suggesting that ferrous hydroxide was converted by oxygen to magnetite at a distance from the metal. Such a conversion would prevent the liquid from being kept saturated with ferrous hydroxide at all points, which would lead to the film being discontinuous. Most of the solid corrosion product was formed near the water-line, and sufficient of it clung to the metal to cause a gain in weight; however, this product seemed to adhere at a few points only, and its removal by pickling revealed deep grooving of the steel below the scale at the water-line zone, and sometimes pitting at points lower down. These observations strongly support the view that the damage caused by oxygen must be attributed to the manner in which it causes the magnetite to be precipitated at a sensible distance from the surface instead of forming a protective film over it.

M/100 sodium chloride or sulphate solution produced much loose corrosion product; after pickling, the loss of weight was greater than that produced by water, but was less localized, extending well above the water-line. The acceleration of attack by salts is evidently due to the formation of freely soluble anodic and cathodic products (*e.g.*, ferrous chloride and sodium hydroxide) which interact out of contact with the metal giving non-protective rust or magnetite, according to the amount of oxygen locally available; in neither case will there be any stifling of corrosion.

M/100 magnesium sulphate seemed to cause a flaky deposit on the metal, which in the 76-day experiments, peeled off above the water-line. The behaviour may be correlated with that at ordinary temperature as follows. In cold magnesium sulphate,[4] the cathodic product magnesium hydroxide, being sparingly soluble, is deposited upon the metal at the cathodic zones. The soluble anodic product, ferrous sulphate, converts it *in situ* to a bright green body (probably hydrated magnetite with part of the Fe'' replaced by Mg''). At low temperatures this is subsequently converted by oxygen *in situ* to a pale clinging form of rust, but at high temperatures it suffers dehydration instead of oxidation, giving a dark coat of flaky magnetite which is protective at first, but ultimately peels off (probably as a result of stress arising from the considerable volume changes involved in dehydration). This may explain the contrast between the behaviour of magnesium salts in hot and cold water.

Alkali diminished the attack by water under aerobic conditions, evidently by depressing the solubility of ferrous hydroxide, thus increasing

the probability of the formation of a continuous film over the surface. $M/100$ sodium hydroxide produced less change of weight than distilled water, whilst $M/10$ alkali left the fully immersed area almost unchanged in appearance after four days, although there was a little etching at the water-line and interference tints above it.*

Thus under aerobic (in contrast with anaerobic) conditions, alkali is beneficial and salts generally harmful. The losses of weight recorded after pickling are very much greater than those suffered under anaerobic conditions.

Intensified Attack due to Oxide Scale

Turner[14] has described how severe pitting or grooving occurs in boilers at interruptions in the mill scale. To examine this intensification, specimens were heated in air to develop oxide films of thickness corresponding to late interference tints. When cool they were engraved with scratch-lines and subjected to boiling $M/100$ sodium chloride or sulphate solution under aerobic conditions. The oxide scale prevented corrosion where intact, but serious attack occurred at the scratch-lines, with deep pits penetrating a considerable fraction of the thickness after 4 days. On the immersed area the penetration was locally deeper than if there had been no scale present, doubtless due to the dangerous combination of the large cathode (the scale) and the small anode (the steel exposed at the scratch-line).

It seemed possible that the dark layer produced by magnesium sulphate might have acted like the oxide film produced by heating in air. To test this, two-stage experiments were carried out. Specimens were first heated in magnesium sulphate solution, the layer was next engraved with a scratch-line, and the specimen was then heated in a salt solution. Various salts were tested and both aerobic and anaerobic conditions included. No intensified attack was obtained, and it was tentatively concluded that, whilst oxide films formed by air heating can certainly produce intensification of attack at discontinuities, the layers left by magnesium sulphate are not dangerous in this respect.

Intensified Attack due to Copper

Boiler water often contains copper salts owing to the use of copper alloys in condensers, pumps, feed-water heaters, and other equipment. It has been suggested that extra corrosion may be caused by copper salts acting on iron to give iron salts and metallic copper. This replacement, however, need not in itself involve additional attack, since copper will in general only pass into the water if oxygen is present, and a given amount of oxygen will cause the same destruction of steel whether (*a*) it comes directly into contact with the steel or (*b*) it acts on copper producing copper salts which subsequently attack the steel. On the other hand, the deposition of metallic copper as a discontinuous layer on the steel might subsequently stimulate attack, as a result of the cell copper–iron, or in other ways.

To test this, steel was exposed to hot $M/100$ copper sulphate, then engraved with a scratch-line, and subsequently exposed to a salt solution under aerobic conditions. Much less action was observed than had been anticipated, and sometimes metallic copper seemed to be redeposited on the scratch-line. Much magnetite and some rust, however, appeared, and the bulging masses of magnetite formed on the copper-covered part of the surface suggested that the decomposition of ferrous hydroxide to magnetite and hydrogen had been catalysed by metallic copper, and that if the area of bare steel were greater, the preferential deposition of magnetite on the coppered portion might hinder the covering-up of the exposed steel with a protective film.

Accordingly, steel specimens were coated over only half of their breadth with copper by immersion in cold $M/1000$ copper sulphate (the amount of iron lost in the replacement, about 0·4 mg., was neglected, being far less than the losses normally suffered in the experiments). The half-coppered specimens were then exposed to boiling water or salt solution, uncoppered specimens being tested for comparison. In anaerobic experiments lasting 6 days, distilled water produced much more corrosion on the half-coppered specimens than on the uncoppered ones; since attack was concentrated on half the area, the intensification was greater than the figures recorded in Tables I and II may seem to indicate. A black varnish-like deposit on the coppered half suggested that the preferential catalytic deposition of magnetite on the copper may have kept the solution over the iron unsaturated with ferrous hydroxide, preventing film-formation. In 6-day aerobic tests in water, half-coppered specimens lost more weight than uncoppered ones; in sodium sulphate there was no significant difference: in both liquids, however, there was intensification of attack near the junction, where the copper–iron cell would operate, leading to pitting or grooving (pitting,

* The corrosion produced by concentrated alkali under stress conditions has not been studied in the present research, but has been discussed by Desch and other workers[11] from the National Physical Laboratory; by Schroeder and Berk,[12] and more recently by Colbeck, Smith, and Powell.[13]

however, sometimes occurred even in experiments where no copper was present).

The 6-day experiments seemed to suggest that copper deposition might be dangerous, but in experiments lasting 76 days half-coppered specimens fared hardly worse than uncoppered specimens, the metallic copper having apparently disappeared. Thornhill[15] has suggested that, although in acid or neutral solutions copper is effectively cathodic to iron, this may not be the case in alkaline solutions where iron tends to become passive and copper can pass into solution as complex anions. Recent values of equilibrium electrode potentials published by Feitknecht,[16] and by Pourbaix,[17] seem to be consistent with this view. The pH value of boiling water in contact with iron rises owing to the formation of ferrous hydroxide, and it is not impossible that a state may be reached where iron is incapable of depositing metallic copper, and where metallic copper already deposited will return to the solution.

Boiler water is normally kept alkaline, and although this may increase the risk of attack on copper fittings if the pH value becomes excessive (*cf.* results of Tronstad and Veimo,[18] and of Akimow and Rosenfeld[19]), it will diminish the chance of enhanced attack on the steel in presence of copper compounds. If locally and temporarily the water became acid, deposition of metallic copper might occur, and if the geometrical distribution were such as to produce the combination of a large cathode (a coppered area) surrounding a small anode (bare steel), the danger of severe pitting might arise. Certain cases of intense pitting or grooving in power boilers are known where compact adherent metallic copper has been found interleaved between magnetite. Further investigation is needed, but it may be tentatively suggested that copper compounds in the water do not produce serious corrosion where water conditions are normal, but that they increase the potential danger that intense attack might arise if, locally and temporarily, water conditions should become abnormal.

Summary of Main Conclusions

Under anaerobic conditions the corrosion of steel by boiling water is of the hydrogen-evolution type ; it soon stifles itself by the formation of a film, which is first probably ferrous hydroxide but is converted *in situ* to magnetite by interaction with water. Sodium hydroxide in the water does not significantly diminish the corrosion rate, whilst sodium chloride or sulphate does not seriously increase it.

Under aerobic conditions, serious local attack occurs near the water-line, evidently because here ferrous hydroxide is converted by oxygen uptake to magnetite or rust *out of physical contact* with the metal, so that a continuous protective film over the steel is not maintained. Sodium hydroxide diminishes the attack (doubtless by depressing the solubility of ferrous hydroxide), whilst sodium chloride, sodium sulphate, or magnesium sulphate increases the attack, which in the sulphate solutions tends to occur mainly above the water-line.

Oxide films produced by heating in air cause intensification of attack at discontinuities in the film, owing to the combination of a large cathode (the oxide) and a small anode (the steel exposed).

Metallic copper deposited over half of the surface increases the intensity of attack in short-time experiments but seems to have little effect in long experiments, where the metallic copper disappears.

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The Determination of the Equilibrium Constant of the Reaction between Molten Iron and Hydrogen Sulphide*

By J. White, D.Sc., Ph.D., A.R.T.C.,†
and H. Skelly, Ph.D.‡

Synopsis

Determinations of the equilibrium constant of this reaction at temperatures above the melting point of iron have previously been made by Maurer and Bischof and by Chipman and Ta Li, the values obtained by the former workers being approximately 100 times greater than those obtained by the latter.

Attempts to determine the value of the constant were made by two different methods, viz., (1) by the use of a high-temperature balance and (2), in a modified apparatus in which four small beads of metal were subjected simultaneously to the action of the gases in a reaction chamber small enough to be contained entirely in the hot zone of the furnace. The first method proved unreliable, owing to thermal diffusion of the gases and excessive sulphur deposition, and the results obtained by the second method are considered the more accurate. The values obtained are, on the whole, somewhat lower than those by Chipman and Ta Li. It is suggested that the true values may be still lower, as it was found that considerable amounts of silicon were picked-up by the metal samples during the runs, appearing to raise the value of the constant. There was evidence that considerable loss of silicon occurred in the H_2S–H_2 atmosphere, from the refractories used in the hot zone of the furnace, this apparently being due to the formation of a volatile silicon compound.

Introduction

This investigation was started just before the outbreak of war and as a consequence has been considerably interrupted by other work, whilst the construction of suitable apparatus has been somewhat hampered by the difficulty in obtaining special materials. In addition, the later part of the work was somewhat curtailed owing to impending staff changes.

Previous Investigations

The equilibrium constant can be expressed as either :

$$K = \frac{H_2S}{H_2} \times \frac{Fe}{FeS} \text{ or } K' = \frac{H_2S}{H_2} \times \frac{1}{S}.$$

In the first form H_2S/H_2 is the volume ratio of the gases at N.T.P., and Fe and FeS are best expressed as molecular concentrations. In the second form the H_2S/H_2 ratio is expressed in the same way, but the sulphur concentration is expressed as a weight percentage and the iron concentration is taken as unity as long as the sulphur concentration is low. Throughout the paper the second expression is used but the data given can readily be converted to either form.

Previous attempts to determine this constant for the case of liquid iron have been made by Maurer and Bischof[1] and by Chipman and Ta Li.[2] The former workers continuously circulated a gas of known composition in the space above the molten charge of steel in a closed crucible, using a Tamman furnace. Whether the sulphur content of the gas increased or diminished depended upon the concentration and also its relation to the equilibrium value. At the end of a given time a portion of the gas was withdrawn for analysis to determine in which direction its composition was changing towards equilibrium. Thus, from the initial composition and the final composition, the direction of the reaction could be ascertained. These authors state that it was impossible to prolong the operation to the point of complete equilibrium, as they found that at the high temperatures used, the crucible and reaction tubes

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† The Refractories Department, Sheffield University ; formerly at the Royal Technical College, Glasgow.

‡ Formerly at the Royal Technical College, Glasgow.

were incapable of resisting the chemical attack of the reacting substances for more than 60 min. The crucibles were made of beryllium oxide and the reaction tubes of 'Pythagoras' material. The melts were made from pure iron and iron sulphide, and the sulphur contents ranged from 0 to 29·6%, the weight of each melt being in the order of 5 to 6 g.* Chipman and Ta Li used an induction furnace to heat their samples of 50 to 80 g. of metal contained in suitable refractory crucibles which were enclosed in a silica tube of about 2 in. outside dia. Three types of crucible were used, magnesite, porcelain, and silica. Magnesite was found to absorb appreciable quantities of H_2S and thus caused errors by altering the gas composition. Silica was apparently the most satisfactory from this point of view, though the silica crucibles sagged rather badly at high temperatures. To prevent errors which may occur due to thermal diffusion when a mixture of gases of different densities are heated in an enclosure in which temperature gradients exist, and which results in a concentration of the heavy gases in the cooler portions of the enclosure, the incoming gases were preheated by passing them over heated molybdenum filaments. Fontana and Chipman[3] had previously found this device effective in preventing thermal separation of hydrogen and steam in studies of the system Fe–H–O. The practical importance of this effect and its influence on the values of the equilibrium constants determined in the latter system have also been demonstrated by the work of Emmett and Shultz,[4] and Eastman and Ruben.[5] By preheating the gases in this manner Chipman and Ta Li claim to have overcome this effect, and the analyses of their outgoing and ingoing gas mixtures once equilibrium had been attained show reasonably good agreement One of their chief difficulties lay in the lengthy period required for the liquid iron to reach a state of equilibrium with the gas mixture. They found that this period was, however, considerably shortened by making up a series of iron–sulphur alloys from which samples of suitably chosen sulphur contents could be made up and used as starting materials. When equilibrium had been reached, as shown by the near agreement of incoming and outgoing gas compositions, the sample was cooled quickly by simply switching off the current (when rapid cooling occurs with an induction furnace). Unfortunately, there is a considerable discrepancy between the values of the equilibrium constant obtained by Maurer and Bischof and those obtained by Chipman and Ta Li, the values of the former workers being approximately 100 times greater than those of the latter, who suggest that the beryllia crucibles used by Maurer and Bischof may have been active in absorbing sulphur. It was because of this discrepancy that the present investigations were undertaken.

1—Investigations Using High-Temperature Balance

The Apparatus

A vertical molybdenum-wound tube furnace, operating in a protective atmosphere of hydrogen and nitrogen obtained by cracking ammonia gas was used throughout. The reaction tube which had to be impervious to gases at high temperatures was long enough to allow its being closed at top and bottom by rubber bungs. In the early part of the work 'Pythagoras' tubes of 5 cm. diameter by 100 cm. long were used, but later these became unobtainable. At about the same time, however, 'Morganite' tubes (Triangle H5) of this size became available and were found to be highly satisfactory and noticeably more refractory than the 'Pythagoras' tubes.

The initial series of experiments was carried out with the crucible containing the melt suspended from the pan of an assay balance placed on a shelf above the furnace (the so-called 'high temperature balance'), the arrangement being as shown in Fig. 1. It was hoped by this means to record the changes in weight due to sulphur pick-up as had been done previously with a similar apparatus in the case of oxygen pick-up by (and evolution from) iron oxide melts. A series of determinations were made using this method but it was later abandoned for reasons given below. To cut down convection currents which would have disturbed the weighings, and to keep the lower bung cool, baffle plates of alundum were placed at suitable levels in the lower part of the reaction tube. These were supported on a central alundum tube through which the 'Pythagoras' thermocouple sheath was threaded, the closed end of the latter projecting about 3 in. above the upper baffle, and being so placed that the thermocouple tip was just below the centre of the hot zone of the furnace. The crucible was arranged to swing just clear of the closed tip of the thermocouple sheath when the balance was at the lowest point of its swing. A platinum/10% platinum–rhodium thermocouple was used, calibrated against the melting points of silver (961° C.), gold (1063° C.), and palladium (1555° C.) using the wire bridge method, and it was checked

* For further details *see* Maurer and Bischof[6]; this gives a more detailed account than the other paper[1] by these workers.

at frequent intervals to ensure that no contamination was occurring. To test the accuracy with which the thermocouple was capable of giving the temperature of the crucible, a forked piece of alundum, across which a piece of palladium wire was laid as a bridge, was lowered into the position normally occupied by the crucible and heated slowly to the melting point of palladium whilst being kept under observation. The melting point obtained agreed closely with the ordinary calibration of the thermocouple.

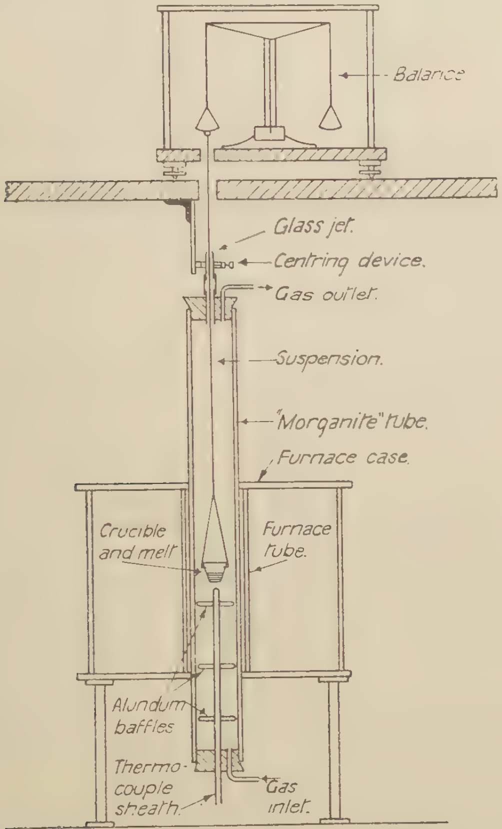

FIG. 1—Arrangement of apparatus incorporating the high-temperature balance

During a run the gas mixture was admitted through a glass tube passing through the lower bung and emerged through a similar tube in the upper bung. In later experiments the direction of gas flow was reversed. The upper part of the suspension which was of fine platinum wire emerged from the reaction tube *via* a glass tube drawn out to a fine jet to minimize loss of gas while a weighing was being made. Between weighings a length of rubber tube, slipped over this glass tube and extending above the jet, was kept tightly clipped on the suspension to prevent gas loss entirely (*see* Fig. 1). This was opened only a few minutes at a time when a weighing was being made. Because of the small degree of clearance between the suspending wire and the sides of the jet, it was essential that the alignment of the latter should be capable of accurate adjustment. This was done by means of three positioning screws threaded through a metal ring situated concentrically with regard to the suspension wire and held in position by means of a bracket screwed on to the shelf supporting the balance. This deviation from the practice adopted when studying iron oxide equilibria with the high-temperature balance (the balance and suspension were totally enclosed to form a continuous closed system with the reaction tube) was necessary as it was obviously undesirable to operate the balance in an atmosphere containing H_2S.

The temperature of the furnace was controlled by a rheostat in series with the furnace winding. A maximum temperature variation of 5° to 6° C. was aimed at during the equilibrium runs and was found to be readily attainable by judicious adjustment of the rheostat.

Crucibles

Alundum crucibles which were made in the laboratory and used throughout as tests, showed a high degree of inertness towards the H_2S–H_2 atmosphere. After forming, the crucibles were first fired to about 1000° C. in an oxidizing atmosphere then, whilst suspended from the balance, they were fired to between 1550° and 1600° C. in hydrogen till the weight became constant.

At the start of this work crucibles treated in this way were afterwards heated in H_2S–H_2 mixtures and the change of weight observed was negligible, though it was observed that the crucible became slightly darker in colour (*see later* concerning possible loss of SiO_2 from alundum in H_2S–H_2 atmospheres). When runs were made with iron in the crucibles, some discoloration of the alundum just above the line of contact between the wall of the crucible and the molten iron meniscus was observed. It was thought that some fluxing of Al_2O_3 by FeS might be occurring, so a crucible in which several runs had been made was broken up. Parts of it were crushed to a powder and boiled with HCl in a flask fitted with an outlet tube which dipped into a solution of ammoniacal cadmium acetate in a boiling tube. No H_2S evolution was observed. Another sample was subjected to fusion and analysed in the ordinary way for sulphur, but again none was detected. It was concluded that any sulphur pick-up must be negligible.

The Suspension

The first suspension used was entirely of platinum wire hooked through holes in the

crucible sides, and from the start trouble was encountered due to its breaking at high temperatures, presumably under the combined effects of the hydrogen and H_2S. The use of thicker wire in the lower part of the suspension effected only a slight improvement in this respect. The use of thin rods of alundum for the lower part of the suspension was then suggested and a number of these were made by extruding alundum mixed with water to a suitable plasticity, through a die of about $\frac{3}{32}$ in. dia. As it emerged the column of alundum was caught on a flat piece of cardboard, straightened out, and while still soft was bent to form a hook at each end. It was then dried and fired to about 1600° C. in a carbon-granule furnace. The rods so made were found to possess ample strength to support the crucible and charge at low temperatures, but were found to break on heating to between 1200° and 1500° C., due apparently to an almost complete loss of tensile strength occurring with softening of the bond. The breaks invariably occurred at the hooks at the lower ends of the rods, so these were abolished. Instead, blobs of alundum were stuck on to the lower ends of the rods which were then made to slide into slotted lugs made on the crucibles. At the same time 3–5% of AlF_3 was mixed with the alundum and the final firing temperature was raised to approximately 1800° C. The rods made in this way were found capable of bearing the load of the crucibles up to 1600° C. They had the disadvantages, however, that they were heavy (two were, of course, necessary to support the crucible) and also that they provided a rather large surface area for sulphur deposition which was found to occur towards their upper (cool) ends causing a change in weight of the suspension. Finally, molybdenum wire of 0·04 in. dia. was used for the lower part of the suspension, two lengths of about 7 in. were hooked at their upper ends to a loop on the end of the platinum suspension, and had hooks passing through holes in the alundum crucible at their lower ends. After an initial heating in an H_2S–H_2 atmosphere these showed little tendency to change in weight apart from a slight but regular gain due to sulphur deposition which, however, was considerably smaller than with the alundum rods.

The Gas System

In this initial series of experiments the gas mixtures were made up and stored at about 1 atm. gauge pressure in a glass carboy of about 45 l. capacity. A two-holes rubber bung was fitted into the mouth of the carboy, cemented with collodion and wired down. One of the connections through the rubber bung led *via* **T**-pieces to a mercury manometer, a Kipp's H_2S apparatus, and a hydrogen cylinder. The other led to the reaction tube and *via* a **T**-piece to a Cenco ‘ Hyvac ’ pump. The various connections were made as far as possible of glass, connected where necessary by short pieces of rubber tubing, so that the area of rubber in contact with the gases was a minimum. A glass tube filled with pumice-stone chips impregnated with copper phosphate was inserted in the system between the carboy and the pump to remove H_2S from the gases before they reached the pump. The H_2S from the Kipp's apparatus was bubbled through water and then over $CaCl_2$ to dry it. Later, silica gel and P_2O_5 were used for this purpose, two drying tubes in series being used. To make up a gas mixture the carboy was first evacuated and a suitable amount of H_2S was admitted from the Kipp's apparatus, the amount being judged from the levels recorded on the manometer. The arm of the manometer to which the H_2S was admitted soon became blackened, so that the level of the mercury could not be seen in it, but this did not matter as the pressure differences could be ascertained by doubling the differences observed in the level in the other arm. Hydrogen from the cylinder was then admitted and the pressure raised to 2 atm. (1 atm. above atmospheric pressure). The gas was then allowed to stand overnight after which its composition was obtained accurately by analysis. For this purpose a sample of the gas was bubbled through ammoniacal cadmium acetate solution to remove H_2S, the remaining hydrogen being collected and its volume measured. The H_2S absorbed was then determined by acidifying and titrating with standard iodine solution, using starch as an internal indicator in the usual way.

Procedure

The iron used throughout the investigation was low-carbon ingot iron. The manufacturers guarantee less than 0·16% total impurities in this material which had the following typical analysis :

C, %	Mn, %	P, %	S, %	Si,
0·012	0·017	0·005	0·025	trace

Fifteen to 20 g. of this material were cut from the bar and placed in a previously weighed crucible which was prepared as described above and heated, suspended from the balance pan, in a stream of hydrogen to a temperature between 1550° and 1600° C. till the weight remained constant over a period of from 30 min. to 1 hr. It was then cooled in hydrogen, and as a check on the accuracy of the weight determined by the high-temperature balance, the crucible and its contents were then weighed on an ordinary balance. The suspension was also weighed before

and after each run so that any change in weight could be allowed for. The weight of the crucible being known, the initial weight of iron in the crucible could be determined. The crucible was then suspended from the balance pan again, and heated to the chosen experimental temperature in a stream of H_2S–H_2 mixture from the carboy. This temperature was maintained, and weighings were made periodically to determine how the pick-up of sulphur by the metal was proceeding.

After the first run it was realized that the time taken to reach equilibrium was going to be excessive, necessitating the use of much more gas than could conveniently be stored. Samples of iron containing sulphur were, therefore, made up by melting fresh iron in a much stronger concentration of H_2S than was used for the actual equilibrium determinations. As the work proceeded a series of samples with different sulphur contents became available and these were used to make up samples of any desired sulphur content. Actually, once an approximate idea of the value of the constant was obtained, a sulphur content was chosen so as to be not too far removed from equilibrium with the gas mixture to be used. Samples both richer and poorer in sulphur than the final equilibrium concentration were made up, so that the equilibrium could be approached from both directions.

At the end of a run the sample was cooled rapidly by pulling the crucible up to the cold end of the tube, and when cold, the crucible and suspension were weighed separately on an ordinary balance to check the weights recorded on the high-temperature balance and also to find out what change in weight of the suspension had occurred. This was necessary since, as mentioned above, sulphur deposition on the suspension wires generally occurred, resulting in a slight gain in weight which had to be allowed for in computing the weight of the metal from the high-temperature balance determinations. The temperature range for such deposition under the conditions existing in the tube was evidently very narrow, as the deposit was confined to a narrow band towards the upper cold ends of the molybdenum wires forming the lower part of the suspension. With the low concentrations of H_2S used the gain in weight of the suspension generally lay between 0·0005 g. and 0·0015 g.

As a check, analyses of some of the melts after equilibrium had been attained were carried out. For this purpose it was necessary to drill the sample in various places and mix the drillings to avoid the effects of possible segregation. The various melts used are indicated by the letters *A*, *B*, *C*, etc. In several cases more than one equilibrium determination was made on the one sample at different gas compositions, this being indicated by suffixing the numbers 1, 2, 3, etc., to the melt letter.

Observations

Table I shows the data obtained from what were regarded as the most successful runs made by this method. The sixth column indicates the change in sulphur content recorded during the closing period of the various runs.

In theory, of course, the attainment of equilibrium should have been indicated by the melt attaining a constant weight. In practice such a condition was found to be very difficult to achieve. A complicating factor was, of course, that the suspension continued to gain weight slowly but fairly regularly during each run due to sulphur deposition, so that absolute weight constancy, even at equilibrium, could not be expected (as described earlier, this gain was allowed for when the suspension was weighed after each run). This, incidentally, robbed the balance of what would have been one of its principal advantages.

Another uncertainty associated with the results recorded in Table I arose from the fact that the analyses of the ingoing and outgoing gases always differed somewhat widely. In early runs it was noticed that when a weak concentration of H_2S was used following a run with a stronger mixture, enrichment of the gas in H_2S occurred. To overcome this, the tube with the baffles and thermocouple sheath inside, was cleaned out between runs by heating to high temperatures with pure hydrogen passing. When the tube had been thus cleaned, it was found that a loss of H_2S occurred during the passage of H_2S–H_2 mixtures through the tube, the composition of the emerging gas being rather variable. Hence, for calculation of the values of the constant shown in Table I, the compositions of the incoming gases only have been used. It may be noted that in somewhat similar circumstances Chipman and Ta Li argued that the incoming gas playing directly on to the metal was probably more representative of the equilibrium concentration than the outgoing gas.

The necessity of decreasing the sulphur deposition was evident, but it was realised that there was little hope of doing so while using the balance, as any increase in the complexity of the apparatus would have increased the difficulty of making the weighings. Again, though the preliminary tests had shown the alundum crucibles to possess, apparently, a high degree of stability, some uncertainty as to their complete weight constancy was still felt and it was realised that a serious disadvantage of the method was that any change

in the weight of the crucible would, of necessity, appear as a change in the sulphur content of the metal. The determination of sulphur in the metal by analysis introduces no such uncertainty, though segregation may, of course, produce errors as pointed out by Chipman and Ta Li. It was, therefore, decided to abandon the use of the balance.

Table I—*Equilibrium Data with High Temperature Balance*

Run	Temperature, °C.	Wt. of Iron, g.	Final Wt. of Iron + Sulphur, g.	Wt. of Sulphur of Iron at Equilibrium, g.	Change in Sulphur Content in Final Period of Run, %	Sulphur by difference, wt.-%	Sulphur by analysis, %	Ingoing Gas Composition. $\frac{H_2S}{H_2} \times 10^2$ (by volume)	$K = \frac{H_2S}{H_2} \times \frac{1}{S(wt.-\%)}$
*A*1	1530	16·9933	17·1333	0·1400	+0·0050 in 110 min.	0·817	0·810	0·293	0·00359
*B*1	1530	17·2400	17·4204	0·1804	+0·0040 in 90 min.	1·036	...	0·466	0·00450
*B*2	1530	17·2400	17·3285	0·0885	−0·0026 in 210 min.	0·511	...	0·217	0·00425
*B*3	1530	17·2400	17·2877	0·0477	+0·0060 in 180 min.	0·276	...	0·113	0·00410
*C*1	1550	20·1230	20·2600	0·1370	+0·0030 in 30 min.	0·676	...	0·277	0·00410
*C*2	1550	20·1230	20·1683	0·0633	+0·0050 in 60 min.	0·314	0·320	0·115	0·00366
*D*1	1550	15·1861	15·2994	0·1133	+0·0050 in 30 min.	0·741	0·732	0·323	0·00436
*E*1	1570	15·9506	16·0145	0·0639	−0·0010 in 140 min.	0·399	...	0·181	0·00454
*E*2	1570	15·9506	15·9933	0·0427	−0·0010 in 40 min.	0·267	...	0·100	0·00375
*F*1	1570	20·2481	20·3291	0·0810	+0·0025 in 50 min.	0·398	0·400	0·178	0·00447
*G*1	1600	19·8255	19·9496	0·1241	−0·0030 in 60 min.	0·622	...	0·261	0·00420
*G*2	1600	19·8255	19·9601	0·1346	+0·0013 in 30 min.	0·674	...	0·308	0·00457

Table II gives the averages of the values obtained at the various temperatures.

2—Investigations with Modified Apparatus

As a result of the experience gained in the preceding work, it was realized that the primary objectives to be aimed at in designing and constructing a new apparatus were (a), that the effects of thermal diffusion and sulphur deposition should be reduced to a minimum and (b), that the time taken to reach equilibrium should be shortened as much as possible.

With regard to the first of these objectives it was decided that the ideal arrangement would be to have the reaction chamber so small that it could be entirely contained within the hot zone of the furnace at a uniform temperature throughout, so that neither thermal diffusion nor sulphur deposition could occur in it. The inlet and outlet tubes to such a chamber, it was considered, should be narrow relative to the diameter of the chamber. Thus, the relatively rapid rate of flow of the gas mixture along such tubes would decrease the tendency for thermal diffusion to occur as the gas was passing along them, while the small inlet and exit openings presented to the gases actually in the chamber at any time would decrease the opportunity for thermal diffusion to occur from the chamber into the tubes. Furthermore, it had

Table II—*Average Values of K at various Temperatures*

Temperature, ° C.	K
1530	0·00411
1550	0·00404
1570	0·00425
1600	0·00439

been noted during the earlier part of the work that sulphur deposition occurred only in two narrow bands inside the long reaction tube then used, indicating a rather narrow temperature range for such deposition. With narrow tubes the

amount of surface presented within this critical temperature range would be small, while as far as the inlet tube was concerned the fact that the gases passed quickly through this zone would give less opportunity for such deposition. At the same time it was realised that too rapid a rate of flow along the outlet tube might not allow time for recombination of the dissociated H_2S before the gas was cooled down too far for recombination to occur. Actually, it was noticed that with rapid rates of flow the outgoing gas usually took on the appearance of fume, possibly owing to finely divided sulphur (the presence of a volatile silicon compound may also have contributed to this, however, as indicated later). The gas flow was, therefore, usually kept below the rate at which the fuming became marked.

It may be noted that this solution of the joint problems of thermal diffusion and sulphur deposition differs somewhat from that used by Chipman and Ta Li, and Chipman and Fontana, as their reaction chambers were tubes passing through the winding of an induction furnace and were cool at the ends. The incoming gases which were led directly to the hot zone and discharged over the metal sample were preheated in their passage down a central tube. This was found effective in cutting down thermal diffusion, presumably by eliminating the chilling action which the introduction of cold gases into the hot zone would have caused. Incidentally, the fact that the incoming gas was discharged directly into the hot zone would produce a still pocket of gas in the upper cold end, thus tending to prevent any rapid flow of the incoming gas to the cold upper end where sulphur deposition would have occurred. There is apparently no reason why sulphur deposition should not have occurred in the lower part of their tube, however. With the present apparatus some sulphur deposition did occur in the outlet tube several inches below the hot zone, but it could not have been large relative to the flow of gas through the furnace, as good agreement was now generally obtained between the ingoing and outgoing gas compositions.

The ideal apparatus would have been one of which the inlet tube, reaction chamber, and outlet tube were constructed entirely of material impervious to the passage of gases and at the same time able to withstand the temperatures and atmospheres used. Such an apparatus would have had to be specially made with special materials and would in war-time have been difficult to obtain. Further, the problem of sealing up the reaction chamber after introduction of the metal samples had to be considered and, as was indeed found by experience, any endeavour to make a cemented seal generally resulted in breaking the apparatus when an attempt was made to re-open the chamber after a run, to remove the metal samples.

The Modified Apparatus

The apparatus depicted in Fig. 2 represents a compromise which was found to be effective in preventing all but a rather small loss of H_2S from the gases passing through. In this apparatus the inlet and outlet tubes were two 'Morganite' (Triangle H5) thermocouple sheaths of about $\frac{1}{4}$-in.

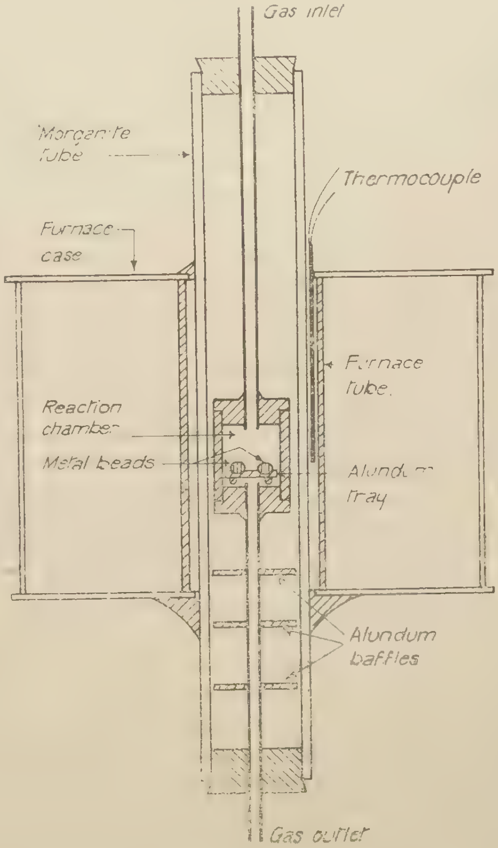

FIG. 2—Modified form of apparatus, showing construction of reaction chamber with inlet and outlet tubes

internal dia. with the closed ends removed. These were impervious to gases. In the first form of the apparatus tried the cylindrical wall of the reaction chamber was made from a length of Morgan 'Triangle H' tube which happened to be available, its internal diameter being about 1 in. The end pieces were made of alundum formed and fired on the ends of the inlet and outlet tubes and recessed to fit as tightly as possible on to the ends of the 1-in. tube. During assembly a wash of alundum was painted over the joints between the

latter tube and the alundum ends. This apparatus had two faults (i), the joints thus cemented could not readily be parted after a run and, as mentioned above, the apparatus generally broke during attempts to dismantle it and (ii), the 'Triangle H' tube blackened and blistered considerably in the H_2S atmosphere and on some occasions was found to be split after a run, due to its tendency to contract, this being resisted by the inserted alundum ends. Alundum was, therefore, used for the body of the reaction chamber, a thick-walled tube (to counteract the porosity of this material) being constructed for the purpose. At the same time the practice of cementing the joints between the tube and the ends was discontinued, the ends being deeply recessed, as indicated in Fig. 2, and ground into the tube to give as tight joints as possible. All the alundum parts were made as impervious as possible by using a high percentage of fine material in making up the plastic mixture. Actually, with these modifications there was no detectable increase in the sulphur deposition over that observed with the cemented form of the apparatus. Finally, all runs were carried out with the apparatus inserted inside the large 'Morganite' tube previously used as a reaction chamber, as shown in Fig. 2, the tube being closed at top and bottom by rubber bungs through which passed the inlet and outlet tubes. During a run this larger tube would, by diffusion through the alundum, become filled with an H_2S–H_2 mixture at the same total pressure as that inside the reaction chamber and, though the gas in the outer tube would tend to become weaker in H_2S due to thermal diffusion and sulphur deposition, it was thought that diffusion of H_2S through the alundum might remain slow, at least relative to the rate at which fresh gas was passing through the tube. This was confirmed by the good agreement obtained between the compositions of the ingoing and outgoing gases.

With the modified form of the apparatus a new arrangement of the thermocouple was adopted for simplicity in assembly. Instead of the platinum/platinum–rhodium a tungsten/molybdenum thermocouple was used enclosed in a sheath of wrought molybdenum to screen it from the field of the furnace winding, and bound to the outside of the large 'Morganite' tube at the level of the metal samples in the reaction chamber. The thermocouple was thus operating in the mixed hydrogen–nitrogen atmosphere of the furnace. It was necessary, of course, to calibrate this thermocouple for temperatures inside the tube, and this was done by suspending a standardized platinum/platinum–rhodium thermocouple inside the tube with air passing through, and comparing the readings obtained on the two thermocouples at various temperatures after the furnace temperature had been held constant for a considerable period. The calibration curve thus obtained gave the temperatures inside the tube under conditions of soaking such as were used during actual experimental runs, but of course, the calibration would not have held for conditions of rising or falling temperature.

Perhaps the greatest innovation now adopted was in the size and weight of the metal samples. It was realized that if the samples were made small enough, they would, due to the high surface tension of the metal, tend to form beads when melted. Such beads could be contained in relatively shallow depressions in an alundum disc, and the bulk of the bead would then sit upon the disc with about two thirds of its surface in free contact with the gases. In addition, with such a small amount of metal the attainment of equilibrium would be very much quicker owing to the smaller weight of sulphur to be picked-up and the short distances involved in the diffusion of the sulphur to the centre of the bead. It was also intended to utilize the whole weight of the bead for the analysis of the sulphur by the gravimetric method (precipitation as $BaSO_4$). For this purpose a bead weighing $0{\cdot}5 - 1{\cdot}0$ g. was found to be ample. Later, when an apparatus for sulphur determination by combustion became available and was put into use, only about $\frac{1}{3}$ of each bead was required, a whole bead containing too much sulphur for convenience. The beads were then broken up by crushing them in the vice, this being comparatively easy owing to their brittleness. Having established that small beads could be used, it was quickly realised that by using more than one bead at a time in the reaction chamber the results could be used to check one another, and that if some of the beads initially contained sulphur while others did not, a criterion that equilibrium had been reached would be obtained if all the beads at the end of a run had similar sulphur contents. Actually, four beads were placed in the apparatus at a time, an alundum tray with four depressions to hold them being constructed for the purpose (*see* Fig. 2). Two of these beads were formed from fresh low carbon iron and two from the sulphur-containing samples obtained in the earlier work with the balance. Owing to changes of personnel in the course of the work the identities of most of these older samples were lost, but this did not matter greatly as long as two of the samples actually did contain sulphur initially, whilst two did not. It will be seen from Table I, however, that the sulphur contents of the former would be between $0{\cdot}267$ and $1{\cdot}036\%$.

The earlier experiments had been considerably handicapped due to the fact that the carboy then used was capable of storing only a limited amount of gas mixture. For these experiments, therefore, a hydrogen cylinder of 40 cu. ft. capacity was used, the procedure being to evacuate the cylinder, admit the necessary amount of H_2S as judged by the manometer, and finally to make up the pressure to approximately 500 lb./sq. in. with hydrogen from a cylinder of 150 cu. ft. capacity. The rates of flow used were such that this amount of gas was used up in the times recorded in Table III.

In making a run the metal samples were first of all heated to temperature in a current of hydrogen. At the chosen temperature above the melting point of iron, the hydrogen stream was cut off and the flow of the gas mixture was started, an initial rapid rate being maintained for a few minutes to sweep out the hydrogen. Thereafter a slower, steady flow was maintained for the duration of the experiment. At the end of the run the gas

TABLE III—*Summary of Data from the Runs Made with the Modified Apparatus*

Run	Material	Duration of Run, hr.	Temperature, °C.	Wt. of Bead, g.†	Sulphur in Bead at End of Run, wt.-%	Method of Analysis	Gas Composition $\frac{H_2S}{H_2} \times 10^2$ (by volume) Ingoing	Outgoing	$K = \frac{H_2S}{H_2} \times \frac{1}{S(\text{wt.-\%})}$ Ingoing Gas	Outgoing Gas	Silicon in Bead at End of Run, wt.-%
I (1)	Low carbon iron	3·5	1555	0·5975	0·207	Gravimetric	0·0498	0·0474	0·00241	0·00229	n.d.
(2)	,, ,,	,,	,,	0·6145	0·163	,,	,,	,,	0·00306	0·00291	,,
(3)	,, ,,	,,	,,	0·5180	0·193	,,	,,	,,	0·00258	0·00246	2·07*
(4)	,, ,,	,,	,,	0·4635	0·178	,,	,,	,,	0·00280	0·00266	2·46*
II (1)	Low carbon iron	6·0	1555	0·8365	3·40	Gravimetric	0·712	0·707	0·00209	0·00208	n.d.
(2)	,, ,,	,,	,,	0·5365	3·430	,,	,,	,,	0·00208	0·00206	,,
(3)	,, ,,	,,	,,	0·5830	3·41	,,	,,	,,	0·00209	0·00207	1·12
(4)	,, ,,	,,	,,	0·5335	3·35	,,	,,	,,	0·00213	0·00211	n.d.
III (1)	Fe-S Alloy	7·0	1555	0·8840	0·848	Gravimetric	0·363	0·360	0·00428	0·00425	2·54*
(2)	,, ,,	,,	,,	1·0730	0·850	,,	,,	,,	0·00427	0·00424	2·65*
(3)	,, ,,	,,	,,	0·6660	1·47	,,	,,	,,	0·00247	0·00245	2·38*
(4)	,, ,,	,,	,,	0·7970	1·23	,,	,,	,,	0·00295	0·00293	2·31*
A_{43} (1)	Fe-S Alloy	7·25	1555	...	0·737	Combustion	0·298	0·282	0·00404	0·00383	n.d.
B_{43} (1)	Low carbon iron or Fe-S alloy	6·5	1555	...	Analysis spoiled						
(2)	,, ,,	,,	,,	...	1·16	Combustion	0·342	0·321	0·00295	0·00277	2·08
(3)	,, ,,	,,	,,	...	1·12	,,	,,	,,	0·00305	0·00287	2·02
(4)	,, ,,	,,	,,	...	1·11	,,	,,	,,	0·00308	0·00289	n.d.
C_{43} (1)	Low carbon iron	6·66	1555	...	1·66	Combustion	0·483	0·376	0·00291	0·00226	1·65
(2)	,, ,,	,,	,,	...	1·37	,,	,,	,,	0·00353	0·00274	n.d.
(3)	Fe-S Alloy	,,	,,	...	1·34	,,	,,	,,	0·00360	0·00281	1·52
(4)	,, ,,	,,	,,	...	1·57	,,	,,	,,	0·00308	0·00239	n.d.
D_{43} (1)	Low carbon iron	6·33	1555	...	0·56	Combustion	0·204	0·187	0·00364	0·00334	1·74
(2)	,, ,,	,,	,,	...	0·57	,,	,,	,,	0·00358	0·00328	n.d.
(3)	Fe-S Alloy	,,	,,	...	0·53	,,	,,	,,	0·00385	0·00353	n.d.
(4)	,, ,,	,,	,,	...	0·59	,,	,,	,,	0·00346	0·00317	n.d.
E_{43} (1)	Low carbon iron	6·42	1555	...	1·13	Combustion	0·315	0·329	0·00279	0·00291	2·10
(2)	,, ,,	,,	,,	...	1·10	,,	,,	,,	0·00286	0·00299	n.d.
(3)	Fe-S Alloy	,,	,,	...	1·15	,,	,,	,,	0·00274	0·00286	1·99
(4)	,, ,,	,,	,,	...	1·14	,,	,,	,,	0·00276	0·00289	n.d.
F_{43} (1)	Low carbon iron	6·58	1600	...	1·62	Combustion	0·558	0·542	0·00345	0·00335	2·23
(2)	,, ,,	,,	,,	...	1·58	,,	,,	,,	0·00353	0·00343	n.d.
(3)	Fe-S Alloy	,,	,,	...	1·71	,,	,,	,,	0·00326	0·00317	2·25
(4)	,, ,,	,,	,,	...	1·52	,,	,,	,,	0·00367	0·00357	n.d.
G_{43} (1)	Low carbon iron	7·0	1600	...	3·65	Combustion	0·883	0·877	0·00242	0·00240	1·53
(2)	,, ,,	,,	,,	...	3·69	,,	,,	,,	0·00239	0·00238	n.d.
(3)	Fe-S Alloy	,,	,,	...	3·47	,,	,,	,,	0·00255	0·00253	1·63
(4)	,, ,,	,,	,,	...	3·28	,,	,,	,,	0·00269	0·00267	n.d.
H_{43} (1)	Low carbon iron	6·75	1600	...	2·94	Combustion	0·763	0·784	0·00259	0·00267	1·38
(2)	,, ,,	,,	,,	...	3·03	,,	,,	,,	0·00252	0·00259	n.d.
(3)	Fe-S Alloy	,,	,,	...	3·09	,,	,,	,,	0·00247	0·00254	1·34
(4)	,, ,,	,,	,,	...	3·00	,,	,,	,,	0·00254	0·00261	n.d.
I_{43} (1)	Low carbon iron	6·33	1578	...	2·09	Combustion	0·710	0·703	0·00340	0·00336	2·04
(2)	,, ,,	,,	,,	...	2·27	,,	,,	,,	0·00313	0·00310	n.d.
(3)	Fe-S Alloy	,,	,,	...	2·27	,,	,,	,,	0·00313	0·00310	1·99
(4)	,, ,,	,,	,,	...	2·32	,,	,,	,,	0·00306	0·00303	n.d.
J_{43} (1)	Low carbon iron	6·83	1578	...	3·47	Combustion	0·873	0·817	0·00252	0·00235	1·63
(2)	,, ,,	,,	,,	...	3·68	,,	,,	,,	0·00237	0·00222	n.d.
(3)	Fe-S Alloy	,,	,,	...	3·25	,,	,,	,,	0·00269	0·00251	n.d.
(4)	,, ,,	,,	,,	...	3·42	,,	,,	,,	0·00255	0·00239	n.d.

n.d. = not determined.

* These Si contents were obtained by assuming that the insoluble residue left after solution of the bead was SiO_2. The remaining Si contents were obtained by actually determining the SiO_2 in the residue.

† The weights of the metal beads were determined only in those cases in which the whole bead was weighed for gravimetric sulphur determination. The others were of the same order, however.

stream was cut off and the furnace allowed to cool. No quench was attempted, but tests were carried out under various conditions of cooling, some runs being cooled in hydrogen, some in the atmosphere in the tube at the end of the run, and some in a slow stream of the H_2S–H_2 mixture. No systematic difference in the constant was observable with these different cooling procedures, which is not surprising when the slow speed of adjustment found between iron and the H_2S–H_2 atmosphere, the smallness of the volume of the reaction chamber, and the low H_2S content of the gases are remembered. Comparatively little passage of sulphur from gas to metal would probably be involved in any adjustment during cooling. Periodically during the run, analyses of the ingoing and outgoing gases were made to see if they were remaining constant. The precaution had previously been taken of storing a fairly strong mixture in the gas cylinder for several days, and it was found that once this had been done there was only a slight tendency for the H_2S concentration to drop as the experiment proceeded. Incidentally, if the cylinder was left standing for a few weeks without H_2S in it, it was generally found necessary to repeat this treatment. This on the whole agrees with the findings of Chipman and Ta Li though they claim to have achieved absolute constancy of the gas mixture once their cylinder was completely saturated.

To allow for a reasonable mixing of the gases before their use they were generally stored overnight, the cylinder being left on its side and turned occasionally till it was put into use by placing in a vertical position. On two occasions some segregation of the gases appeared to have occurred, when a rise in the H_2S concentration of the gas occurred towards the end of the run. Table III summarizes the data for the various runs made with the re-designed apparatus.

It was found at the end of Run A_{43} that the four small beads had coalesced into one large one. It is probably to be expected that the values of the constant for such a large bead would be high.

Run C_{43} was anomalous in that the H_2S content of the ingoing gas rose from 0·427 c.c. per 100 c.c. of hydrogen, to 0·483 c.c. (the value recorded in the table) during the last hour of the run. The values of the constant given in the table are, therefore, probably high. An hour before the end of the run the gas analyses were 0·427 (ingoing) and 0·377 (outgoing), giving the constant as 0·00257 and 0·00227 (sample 1), 0·00312 and 0·00275 (sample 2), 0·00318 and 0·00281 (sample 3), and 0·00272 and 0·00240 (sample 4).

Run F_{43} similarly showed a high H_2S concentration in the gases towards the end. Approximately 1 hr. before the end of the run the gas composition was found to be 0·450 (ingoing) and 0·482 (outgoing). These would give the constant as 0·00278 and 0·00298 (sample 1), 0·00285 and 0·00305 (sample 2), 0·00264 and 0·00282 (sample 3), and 0·00296 and 0·00317 (sample 4).

The gas analyses recorded in the eighth and ninth columns of Table III used for the calculation of the constant, were in each case the last analyses taken during the run, and were always taken within the last 20 min. before shutting off the furnace.

It will be noted (*see* last column) that the silicon contents of a considerable number of the beads were determined. This was done after it had been noted that a fairly bulky white residue was left whenever one of the beads was dissolved in acid. In the early runs this was weighed as insoluble residue, but in all the later runs the silica contents were actually determined by treatment with hydrofluoric acid. In all cases this left only a trace of material other than silica. Special attention was paid to the question of silicon content after the first few runs for, on the basis of these, the opinion was formed that a high silicon content appeared to have a tendency to raise the value of the constant. Some confirmation of this view was obtained on consulting the data of Chipman and Ta Li, for the values of the constant which they obtained with silica crucibles are on the average higher than those with magnesia and porcelain crucibles. They report the silicon contents of three of their melts as 0·41, 0·81, and 0·86%, respectively, but consider that it had probably a negligible effect on the value of the constant. The completed data of Table III is not conclusive on the point, though it appears significant that the lowest values of the constant recorded were obtained with low silicon contents, and the highest with high silicon contents. Incidentally, the silicon contents seemed to vary with the highest temperatures attained during the run. On first melting, to hasten the attainment of thermal equilibrium, the temperature was usually raised somewhat above the selected equilibrium temperature, and there appeared to be a distinct tendency for the silicon content to be highest when a higher temperature than usual had been reached at this stage.

The question of the mechanism of silicon pick-up is of some interest. Chipman and Ta Li reported considerable sublimation of SiO_2 which formed a black deposit at the outlet end of their tube. They appear to consider that it was due to reduction of silica at high temperatures by hydrogen. During the present work with the re-designed apparatus, the outlet tube showed a tendency to

choke and had to be cleared between runs. This was partly due to sulphur depositing in the cold parts of the tube well below the hot zone. Nearer the hot zone, however, a much harder type of deposit which was much more difficult to clear away was formed. At the end of the last run the opportunity was taken of breaking the outlet tube in this region, when it was found to be about half closed by a light-grey deposit most of which had apparently formed during the preceding run, as the tube had been cleared before the run by passing a rod through it. A sample of the grey deposit was collected and was found to have negligible loss on ignition showing free sulphur to be absent, but 98% of it was dissolved away by treatment with hydrofluoric acid, showing it to have consisted almost entirely of silica, the residue appeared to be mainly iron. It would appear, therefore, that the silicon found in the metal beads was carried mainly by the gases in a volatile form. The authors, however, have never noticed heavy deposits of this sort in working with hydrogen atmospheres alone, and it would seem, therefore, that the H_2S must play a part in whatever reaction is involved. In this connection it may be noted that the compounds SiS and SiS_2 have both been described in the literature.[7,8] Both compounds have apparently a high vapour pressure, particularly SiS_2, which is said to sublime readily. An alternative explanation may be suggested in the light of a recent paper by Zapffe[9] on the volatile sub-oxide of silicon, SiO, which is fairly readily formed by the action of powerful reducers such as carbon or aluminium on SiO_2 at high temperatures. If this is the mechanism, however, it is difficult to understand why the addition of H_2S to the hydrogen should make such a difference to the amount of silicon volatilized.

The fact that the Si was redeposited as SiO_2 in the relatively cool part of the tube is of interest, as it indicates that sufficient oxygen had been present to bring about re-oxidation as the temperature dropped, though insufficient to prevent reduction in the hottest zone. As regards the source of the silicon, this would presumably come largely from the inlet and outlet tubes, which were definitely of a siliceous type, possibly approaching mullite in composition. Some might also come from the alundum which would possibly have a small amount of clay admixed with it to confer plasticity. It would appear, therefore, that mixtures of H_2S and H_2 are capable of extracting silicon at sufficiently high temperatures from bodies consisting of alumina and silica.

Just above the zone of silica deposition in the outlet tube a number of very small metallic beads were noticed adhering to the walls of the tube. On testing, these were found to be mainly of iron. They had been formed, apparently, by volatilization of iron from the molten samples.

Discussion of Results

If the average of all the results obtained with the modified apparatus are calculated the following values are obtained :

Temp. °C.	K, (Ingoing)	K, (Outgoing)	K, (Mean)
1555	0·00304	0·00286	0·00295
1578	0·00286	0·00276	0·00281
1600	0·00284	0·00283	0·00284

However, this includes values which are suspect for various reasons as mentioned previously, and if the values derived from runs A_{43} and III (1) and (2) (the latter because the high values obtained may have resulted from the combined effects of the larger bead size and the high silicon contents) are omitted and further, if the altered values for C_{43} and F_{43} given in the remarks on Table III are used, the following average values are obtained :

Temp. °C.	K, (Ingoing)	K, (Outgoing)	K, (Mean)
1555	0·00280	0·00271	0·00276
1578	0·00286	0·00276	0·00281
1600	0·00262	0·00270	0·00266

Actually, the taking of an average is of doubtful validity as a means of obtaining the best value, since it would appear that the errors occurring are mainly those which would tend to give high values of the constant, *i.e.*, low sulphur contents. Hence, there is some justification for taking the lowest values recorded as the best.

The runs giving the lowest average values at the three experimental temperatures were II, J_{43}, and G_{43}, these values being :

Run	Temp. °C.	K, (Ingoing)	K, (Outgoing)	K, (Mean)
II	1555	0·00210	0·00208	0·00209
J_{43}	1578	0·00253	0·00237	0·00245
G_{43}	1600	0·00251	0·00247	0·00249

It is significant that the silicon contents determined on samples from these runs are all low.

The lowest values of the constant from individual melts are :

Melt	Temp. °C.	K, (Ingoing)	K, (Outgoing)	K, (Mean)
II (2)	1555	0·00208	0·00206	0·00207
J_{43} (2)	1578	0·00237	0·00222	0·00230
G_{43} (2)	1600	0·00239	0·00238	0·00239

It can hardly be said that the trend with temperature is shown very conclusively by these results. Chipman and Ta Li, the scatter of whose values is as wide as those shown in Table III, drew mean curves through their values plotted against temperature (this being equivalent to taking an average) and showed the value of the constant to be increasing somewhat with the

temperature. While this appears probable, the scatter of the values at each temperature is still too great to allow the trend with temperature to be conclusively stated, but it is at any rate, fairly certain that the rate of change of the value with temperature over the range studied is small. Unfortunately, the temperature range studied was limited by the necessity of keeping above the freezing point of iron at one extreme, and by the capability of the apparatus to withstand higher temperatures in the type of atmosphere employed, at the other.

As regards the actual values of the constant, a definite impression was gained in the course of the latter part of the work that Chipman and Ta Li's values were too high. This is brought out in Fig. 3, where the values of log K for all the individual beads (each averaged between ingoing and

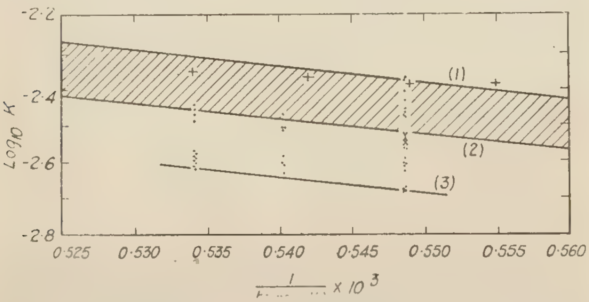

Fig. 3—Comparison of results from the present investigations with those of Chipman and Ta Li

outgoing gas values) are plotted against 1/temp. (° K.). In plotting Fig. 3 all the values given in Table III, bad as well as good, have been included, which is one reason for the considerable spread of the determinations at 1550° C. On the same diagram is shown the band (indicated by the shaded area between curves (1) and (2)), over which Chipman and Ta Li's[2] values are spread. Curve (1) corresponds to their determinations with silica crucibles which they regarded as being their best values. It also agrees closely with the thermodynamic constant for this reaction given by Chipman,[10] these being apparently based on it. Curve (3) is drawn to fit the lowest values obtained in the present investigation, which as already indicated are regarded as being nearest to the true values. The crosses show the averages of the results obtained with the high-temperature balance.

According to Chipman's data (*loc. cit.*), the equation curve (1) is:

$$\log K = \frac{-4500}{\text{temp. (°K.)}} + 0\cdot094,$$

while curve (3) has the equation:

$$\log K = \frac{-4526}{\text{temp. (°K.)}} - 0\cdot20.$$

This gives a thermal value for the reaction which agrees closely with that indicated by Chipman, but makes the entropy change accompanying this reaction of opposite sign to Chipman's equation.

If the effect of silicon is as suspected, the true values of the constant may be still lower than is indicated by curve (3), and in this connection it may be significant that the values indicated by Schenck,[11] based on extrapolation by calculation of data obtained at a much lower temperature by Jellinck and Zakowski,[12] are somewhat lower than those indicated by curve (3) (*e.g.*, K $= 0\cdot0012$ approximately, at 1600° C.). At the low temperatures at which the original determinations by Jellinck and Zakowski were made, silicon pick-up would presumably be small. To settle the matter it would probably be necessary to carry out determinations in an apparatus constructed of materials which are entirely free from silica.

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B. I. S. R. A.

BRITISH IRON AND STEEL RESEARCH ASSOCIATION

A Symposium on the Contamination of Platinum Thermocouples*

By The Liquid Steel Temperature Sub-Committee†

Introduction

The papers included in this symposium demand a brief introduction by reason of the form in which they are presented. During the past four years a number of reports have been presented to the Liquid Steel Temperature Sub-Committee† on the subject of the contamination of platinum thermocouples. These reports form an historical sequence describing the progressive elucidation of a problem of practical importance, and it was felt that in publication, each investigator should receive due credit for his contribution to the co-operative effort. The reports are therefore published substantially as they were presented to the Sub-Committee and should be read as an historical series. The dates of the original reports are given in footnotes to the individual papers.

In the early days of the quick-immersion method, little trouble was experienced with thermocouple failures, but as the use of graphite end-blocks became generally adopted, users began to report occasional outbreaks of thermocouple embrittlement. It is now clear that such embrittlement is due to the sulphur content of traces of oil left in the thermocouple tube, which, in the presence of carbon monoxide, reacts with siliceous materials to form silicon sulphide. The silicon sulphide vapour attacks the rare-metal wires forming one or more of a number of silicides of platinum.

The role of oil in causing the embrittlement was suspected by Land and confirmed independently by Reeve. The work of Chaston established the sequence of reactions causing the embrittlement and identified sulphur as the agent responsible for the transfer of silicon to the thermocouple wires. Goldschmidt and Land identified a number of silicides by X-ray analysis of contaminated wires, and Manterfield found the same silicides in other wires which had failed under rather abnormal conditions. Microscopic examinations of several of the samples, made by Jewell, confirmed the presence of three different contaminant phases.

The practical conclusion is that it is essential to burn out all parts which might be contaminated with oil or other sulphur-bearing materials, particularly if a graphite end-block is used. The necessity of this precaution was foreseen and emphasised by Dr. Schofield when he first introduced the quick-immersion method, but we have now the satisfaction of understanding the mechanism of the process of embrittlement.

Beyond this, however, the results of the investigations suggest alternative combinations of materials which might be satisfactorily used in liquid-steel pyrometry, and new developments will certainly be stimulated.

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* Paper No. 37/1946 submitted by the Liquid Steel Temperature Sub-Committee of the Ingot Committee, received 27th May, 1946.

† The Liquid Steel Temperature Sub-Committee and the Foundry Steel Temperature Sub-Committee of the Iron and Steel Institute were together reconstituted in April, 1946, to form the Pyrometry Sub-Committee of the Steel-making Division of the British Iron and Steel Research Association.

Section I—An Investigation of the Embrittlement of Platinum-Rhodium Wire in the Heads of Liquid-Steel Pyrometers*

By T. Land, M.A., A.Inst.P.†

Synopsis

The embrittlement of platinum thermocouples in liquid-steel pyrometers has been investigated experimentally by exposing new thermocouple wires to different combinations of the materials in proximity to the wire in the pyrometer. It is concluded that oil or other carbonaceous materials in the thermocouple tubes are the cause of the embrittlement.

Serious wire breakages have recently been experienced in the heads of the pyrometers used on the acid open-hearth and basic electric-arc furnaces. The same trouble had occurred before in a less acute form, but had always cured itself without causing undue dislocation of routine control. This time the malady was so severe that experiments were made to isolate the cause. The results are recorded in the present report.

The construction of the pyrometers is as follows:

A length of $\frac{5}{8}$-in. gas pipe bends over at the hot end and carries a graphite block $4\frac{1}{2}$ in. long and $1\frac{1}{2}$ in. dia., which dips into the steel. The gas pipe passes inside the graphite to within $\frac{1}{2}$ in. of the bottom. The silica sheath fits inside the graphite and the steel tube, and overlaps the steel tube by about 2 in. It is held in position with Sairset cement, or alternatively with asbestos string. The gas pipe is insulated with about $\frac{1}{2}$ in. of Sairset cement.

It was noticed that the breakage most frequently occurred at the first gap in the twin-bore silica tubing above the silica sheath. A slight blackening of the wire was often visible immediately round this fracture. The fracture itself was brittle and had a bright crystalline appearance, and no noticeable reduction of area. It was found that the embrittlement of the wire rarely occurred when diatomite bricks were used in place of the graphite blocks.

Method

In order to be able to control the conditions a model of the pyrometer head was made. This merely differed from the pyrometers in that the length of steel tube was only 9 in., and it was not insulated with Sairset cement. Before treatment all wire was annealed at 1200° C. for 5 min. Since the breakages were most frequent in the platinum–rhodium wire, a piece of this about 6 in. long was placed in the assembly so that it reached the bottom of the silica sheath. The complete unit was then placed in an electric-resistance furnace standing at about 1000° C. and left there for a period, usually about an hour.

After treatment the wire was removed and divided into three portions which were tested separately. The test was a simple tensile test in which the load was measured on a spring balance. The type of fracture resulting was then examined. The fractures were of two distinct types; brittle, and ductile. Tests were taken with the full assembly of graphite, steel tube, silica sheath, and Sairset or asbestos. The experiment was then repeated with different combinations of materials omitted, in order to isolate the contaminating factor.

Results

The results are recorded in Table I. Between Tests 7 and 8 the steel tube was heated for $2\frac{1}{2}$ hr. in a gas-fired furnace at 800° C. In Test 12 and subsequent experiments a new steel tube was substituted.

It will be seen that contrary to what might be expected, the graphite did not cause embrittlement. In these tests the wire was laid directly on the inner surface of the graphite block. On withdrawing the block from the furnace, blue flames of burning carbon monoxide played around the ends of the block.

The full assembly, using either Sairset cement or asbestos to secure the silica sheath, gave brittle fractures until the steel tube was very thoroughly burnt out. After that there was only one fracture which showed any signs of embrittlement, although there was a slight reduction of tensile strength on one or two tests. The use of

* Original report submitted to the Liquid Steel Temperature Sub-Committee on 21st April, 1942.

† Research Department, Wm. Jessop & Sons, Ltd., Sheffield.

TABLE I—*Results of Tests*

Test	Materials in Proximity of Wire	Nearest Material	Temperature, °C.	Time, min.	Fracture	Breaking Load, lb.
1	—	— ...	1200	5	Ductile ...	$17\frac{1}{4}$
2	Silica.	— ...	1000	10	Ductile ...	$17\frac{1}{4}$
3	Graphite, steel, Sairset, and silica.	Silica ...	900	15	Ductile ...	$17\frac{1}{4}$
	Graphite, steel, Sairset, and silica.	Metal ...	900	15	Brittle ...	16
4	Graphite.	— ...	900	15	Ductile ...	17
5	Graphite, steel, asbestos and silica.	Silica ...	1000	60	Ductile ...	$17\frac{1}{4}$
	Graphite, steel, asbestos and silica.	Centre ...	1000	60	Brittle ...	$12\frac{1}{2}$
	Graphite, steel, asbestos and silica.	Metal ...	1000	60	Brittle ...	11,* 14*
6	Graphite, steel, Sairset, and silica.	Silica ...	1000	60	Ductile ...	$16\frac{1}{4}$
	Graphite, steel, Sairset, and silica.	Centre ...	1000	60	Brittle ...	15
	Graphite, steel, Sairset, and silica.	Metal ...	1000	60	Brittle ...	10,* 15,* 14*
7	Graphite.	—	1000	60	Ductile ...	17
	Steel tube burnt out in gas furnace					
8	Steel.	—	1000	60	Ductile ...	$17\frac{1}{4}$
9	Steel, Sairset, and silica.	Silica ...	1000	60	Ductile ...	17
	Steel, Sairset, and silica.	Centre ...	1000	60	Ductile ...	16
	Steel, Sairset, and silica.	Metal ...	1000	60	Ductile ...	$16\frac{1}{4}$,* $15\frac{3}{4}$*
10	Graphite, Sairset.	Sairset ...	1000	60	Ductile ...	16
	Graphite, Sairset.	Centre ...	1000	60	Ductile ...	$17\frac{1}{4}$
	Graphite, Sairset.	Graphite ...	1000	60	Ductile ...	$17\frac{1}{4}$
11	Graphite, steel, Sairset, and silica.	Silica ...	1000	60	Ductile ...	17
	Graphite, steel, Sairset, and silica.	Centre ...	1000	60	Ductile ...	$17\frac{1}{4}$
	Graphite, steel, Sairset, and silica.	Metal ...	1000	60	Ductile ...	$17\frac{1}{4}$
	New steel tube					
12	Graphite, steel, Sairset, and silica.	Silica ...	1000	60	Ductile ...	$15\frac{3}{4}$,* $17\frac{1}{4}$*
	Graphite, steel, Sairset, and silica.	Centre ...	1000	60	Brittle ...	$14\frac{1}{2}$
	Graphite, steel, Sairset, and silica.	Metal ...	1000	60	Ductile ...	$16\frac{1}{4}$
13	Steel.	Metal ...	1000	60	Ductile ...	17
14	Graphite, steel, Sairset, and silica.	Silica ...	1000	60	Ductile ...	$16\frac{1}{2}$
	Graphite, steel, Sairset, and silica.	Centre ...	1000	60	Ductile ...	$16\frac{1}{4}$
15	Graphite, steel, Sairset, and silica.	Metal ...	1000	60	Brittle ...	15
	Steel.	Metal ...	1000	16 hr.	Ductile ...	$16\frac{1}{4}$

* Wire broke in shackles

a new steel tube caused a return of the embrittlement which persisted for several hours. Test 13 seems anomalous, as it showed no contamination.

CONCLUSIONS

The embrittlement seems to be caused by the steel tube. Most probably oil and other carbonaceous materials are responsible. This conclusion is borne out by the fact that the trouble with the pyrometers was subsequently cured by very thoroughly burning out the tubes. Heating alone seems to be insufficient, and it is now our practice to heat the tubes and blow a blast of air through them intermittently over a period of an hour. It is also of interest to find that the graphite does not affect the strength of the wire under the conditions of the experiment.

Section II—Fracture of Platinum and Platinum-13% Rhodium Wires used in the Immersion Thermocouple*

By L. Reeve, Ph.D., B.Sc.,† and A. Howard, B.Met.†

SYNOPSIS

A test is described which simulates the intercrystalline attack on platinum and platinum–13% rhodium thermocouple wires by heating a short length in a graphite block in the presence of a small quantity of lubricating oil, the wire being placed in a silica insulator. The test has been standardized at 1400° C. for 15 min., using one drop (0·05 c.c.) of Shell B. 6 oil, which contains 0·2% of sulphur.

It is shown that attack is less marked on fully annealed or fully cold-worked wire than on wire which has been only partially annealed.

Modified oil tests are described which confirm the conclusions of Chaston and his co-workers (described elsewhere in this symposium), that silica and sulphur are essential constituents of the attacking medium.

Spectrographic examinations of attacked wires are reported, which show that copper, silicon, manganese, nickel, and iron (in decreasing order of amount) are present, particularly in the areas of maximum contamination.

Thorough baking at a dull-red heat of the iron pipes used for carrying the thermocouple wires, with a current of air drawn through them, results in almost complete freedom from this type of intercrystalline failure in practice.

INTRODUCTION

THE following experiences with platinum and platinum–13% rhodium wire may be of interest to other users of the Schofield–Grace immersion thermocouple. In the early stages of our work with this instrument we experienced frequent wire breakages, usually of the platinum–rhodium member. These were usually reported to be within 12 in. of the hot junction. Some of these failures, and especially those in the softer platinum members, were due to purely mechanical causes, such as damage to the wire on passing over the sharp edge of insulators; others, however, were found to be due to intercrystalline attack, and a photomicrograph of a typical failure of this type in a platinum–rhodium wire is shown in Fig. 4. This shows clearly the intercrystalline nature of the attack, with the formation of some dark boundary constituent. This particular platinum–rhodium wire was obtained from Batch *A* referred to below, and it failed at a point some 10 in. from the junction after only three immersions. Preliminary reports on these early breakdowns indicated that on the whole the platinum–rhodium wire from Batch *A* was considerably more liable to attack than wire from a second batch, *B*, but in order to arrive at a definite decision as to the cause of these breakdowns and the reasons for the better behaviour of Batch *B*, a series of laboratory tests was carried out on both wires, a résumé of which follows.

LABORATORY TESTS ON NORMAL PLATINUM–RHODIUM THERMOCOUPLE WIRE

As-Received Structure

Wires from both sources showed a uniform polygonal grain structure, wire *B* having a smaller grain-size. Grain-size of *A* was approx. 6, and *B* approx. 7, on the A.S.T.M. scale.

Study of Grain Growth

Tests were carried out at elevated temperatures to determine the degree and rapidity of grain growth. The grain-sizes were related to the heating temperatures and time, but invariably the growth of wire *A* was greater than that of wire *B*, and was also more erratic; large grains often occupied whole cross-sectional areas of the wire and were bordered with colonies of smaller grains. Wire *B* was more homogeneous in its growth. The grain-size of wire *B*, after being maintained at 1350° C. for $1\frac{1}{4}$ hr., was approximately size 3 on the A.S.T.M. scale. Wire *A*, after being heated to 1300° C. for only half an hour showed, despite the lower temperature and shorter time, a grain growth already somewhat greater than that of wire *B*.

* Reported to the Liquid Steel Temperature Sub-Committee, 21st March, 1944.

† Metallurgical Dept., Appleby-Frodingham Steel Co., Ltd., Scunthorpe.

Bending Tests

Samples of the two wires were bent until 'frosted,' and then underwent the following treatments :

(*a*) Micro-examination.

(*b*) Heating in graphite to various temperatures (1370° C. max.), usually for 1 hr., and then micro-examined.

(*c*) Similar to (*b*), except that the wires were sheathed in silica insulating tubing.

It was hoped that by initially disrupting the surface of the wire by the bending, any attack undergone in (*b*) or (*c*) would have a greater effect. It was suspected that repeated bending in the thermocouple might be a cause of early failure.

No conclusive results were forthcoming, however, apart from the already observed fact that the wire *A* showed larger grain growth in all cases.

In one isolated case, after heating samples embedded in graphite for 2 hr. at 1370° C., a light-blue boundary precipitate was noticed in the wire *A*.

Heating in a Reducing Flame

It has been reported that platinum is attacked by heating in highly carburetted gases and vapours. The metal disintegrates, possibly with the formation of PtC_2 and becomes very brittle.

Samples of the two wires were consequently heated in the reducing zone of a coal-gas flame for 2 hr.

Visual examination showed (after removal of a deposit of soot) a brightening of the wire surface, together with etching of the grain boundaries, but micro-examination showed no evidence of intercrystalline attack. It is possible that the conditions of the test for producing this type of attack are fairly critical. In any case the matter was not followed up, as in the meantime promising results were being obtained from a test involving heating in the presence of oil.

Heating in the Presence of Oil

The arrangement for these tests is shown in Fig. 1. The holes are $\frac{3}{16}$-in. dia., and are deliberately sloped as shown.

The oil used was a heavy lubricating grade, Shell B. 6, which contains 0·2% of sulphur; 3 drops (approx. 0·15 c.c.) were placed in each hole in the graphite block at the commencement of each test. A $\frac{3}{4}$-in. length of wire was used in each test, usually in a silica insulator, the last $\frac{1}{8}$ in. of the wire being bent as shown.

An initial test carried out at 1370° C. for $1\frac{1}{2}$ hr. practically disintegrated the bottom $\frac{3}{8}$ in. of the wires. These conditions had been too severe, and subsequently a series of tests of $\frac{1}{2}$ hr. duration were carried out at 1100°, 1200°, and 1300° C., and a further test of $\frac{1}{4}$ hr. duration at 1400° C. In this way it was hoped to arrive at a standardized test which could be used for testing further supplies of wire.

The tests at 1100° and 1200° C., although showing superficial attack and pitting, did not show any serious intercrystalline attack on micro-examination. There were occasional slight inroads of attack at surface grain boundaries (at 1200° C.), but attack was not considered sufficient for comparison between the two wires.

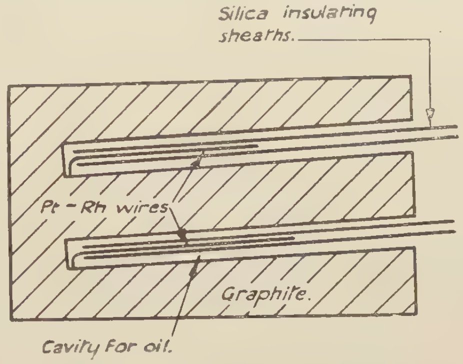


FIG. 1—Assembly used for oil tests

At 1300° C. the results were more decisive, the bottom sections of the wires being subjected to rather severe grain-boundary attack, which penetrated considerably in the case of wire *A*.

The results from the $\frac{1}{4}$-hr. tests at 1400° C. were even more conclusive. Wire *A* was severely attacked, practically complete cross-sectional areas of the wire being disintegrated. Wire *B* also showed some badly attacked areas near the surface only, but little deep penetration.

Figure 6 shows a section of wire *A* almost completely disintegrated. This photomicrograph was taken at the centre of the wire. Wire *B*, as already stated, was only superficially attacked.

In all these oil tests, attack was concentrated mainly at the exposed portion of the wires just outside the silica sheaths. Further tests carried out for $\frac{1}{2}$ hr. duration at 1300° C. without the silica sheaths, resulted in slight general attack all along the wire. This point is of some importance in connection with the causes of attack, and is referred to again later.

Influence of Quantity of Oil

In order to reduce the degree of attack the quantity of oil was then reduced to 1 drop (approx. 0·05 c.c.), sufficient time being allowed before each test to enable the oil to soak into the graphite. On testing at 1400° C. for 15 min.

using this reduced quantity of oil, the results obtained with wires *A* and *B* are shown in Figs. 5 and 8. The degree of attack has been reduced, as would be expected, but wire *A* again shows considerably more attack than wire *B*.

Influence of Pre-Annealing the Wire

Whilst the test as it stood apparently distinguished quite clearly between the two batches of wire, no reason had yet been found to explain this difference. Measurements of e.m.f. carried out with both types of wire gave substantially the same results, whilst a spectrographic examination of both wires indicated substantially the same degree of purity.

The matter at this stage was referred to the suppliers of one of the wires (The Sheffield Smelting Co., Ltd.), who suggested that the difference was probably due to the degree of annealing to which the wires had been subjected. They indicated that, in their view, wire *A*, which had been giving unsatisfactory results, was probably only partially annealed, whilst wire *B* was probably fully annealed. We obtained from them two lengths of platinum–13% rhodium wire of ½-mm. dia., drawn from the same billet, one of which (wire *C*) was in the cold-rolled condition, whilst the other (wire *D*) was fully annealed.

The following examinations were made of these two samples :

(*a*) Structure as-received.
(*b*) Grain growth at 1400° C.
(*c*) Oil attack at 1400° C.

(a) Structure As-Received

Wire C (*cold worked*)—Marked cold-worked structure; extremely elongated grain difficult to resolve at × 120 magnification. Average hardness 211 V.P.N. (1 kg. load).

Wire D (*fully annealed*)—Equiaxed structure similar to that of wire *A* (grain-size 6). Average hardness 132 V.P.N.

(b) Grain Growth, 15 min. at 1400° C. (in silica insulators)

Wire C (*cold worked*)—Grain-size 3, but containing occasional colonies of smaller grains. Hardness values ranged from 153 V.P.N. at the end inside the silica insulator down to 99 V.P.N. at the exposed portion. It is unlikely that this was due to non-uniform heating.

Wire D (*fully annealed*)—Homogeneous grain growth, approximately grain-size 3. Where the wire had been bent at right-angles, substantially larger grains were present, indicating the effect of straining on the resulting grain-size. Average hardness 110 V.P.N., fairly uniform.

(c) Oil Attack. One Drop of Oil, 15 min. at 1400° C.

Both wires were only slightly attacked, the attack being limited to occasional surface penetrations. The degree of attack was similar to that of wire *B* in Fig. 8.

It was concluded that fully annealed and fully cold-worked wires have given better results with less grain growth and less attack by oil than the partially annealed wire *A*. It must be assumed, therefore, that the attack on wire *A* was accelerated by a critical amount of residual strain.

In view of the important conclusions drawn from the oil test, a repeat experiment was finally carried out on lengths of the fully annealed wire *D*, together with a piece of the original partially annealed wire *A*, which were subjected to the oil test simultaneously. This confirmed that the attack on the partially annealed wire *A* was considerably more marked than on the fully annealed wire *D*.

Influence of Sulphur and Silica

Following the issue of the memorandum "Embrittlement of Platinum/Platinum–Rhodium Thermocouples," by J. C. Chaston (reported elsewhere in this symposium), in 1944, the following modifications of the oil test were carried out to confirm the hypothesis that silica and sulphur played a part in the embrittlement.

An oil test was carried out on a sample of platinum–13% rhodium wire *C* with two drops of oil B. 6 in a graphite block for ½ hr. at 1400° C., the wire being in a fused-alumina insulator. No attack could be detected on micro-examination. This was repeated on another batch of fully annealed wire *E* with the same result. Both wires under the same conditions in a silica insulator with one drop of oil showed the usual attack after 15 min. at 1400° C. Silica is thus an essential constituent of the attacking compound.

It was also confirmed that the oil could be replaced by flowers of sulphur with the same results, *i.e.*, attack if in a silica insulator, but no attack if surrounded by fused alumina.

Similarly, the use of pure liquid paraffin instead of Shell B. 6 oil prevented attack even in a silica insulator.

The use of a graphite holder, together with oil or flowers of sulphur, but without the presence of a silica insulator, practically suppressed attack.

Hence the simultaneous presence of silica, sulphur, and probably carbon or graphite are necessary to produce the attack.

Whilst platinum silicide is probably the main intergranular constituent, attention should be

drawn to the results of the spectrographic analyses reported by Mr. Jewell, which show that the major impurity in the embrittled area is not silicon, but copper.

This point needs further attention, since traces of copper are present in many open-hearth-steel furnaces.

Oil Tests on Platinum Wire

All the tests reported in Mr. Jewell's paper have been carried out on platinum–13% rhodium wires, as the intercrystalline failures were originally reported in such wires. Oil tests have since been carried out on pure platinum wire using one drop of oil in a graphite block with a silica sheath. These indicate that a similar type of intercrystalline attack is produced. When sulphur is used instead of oil, the attack is very marked. In actual practice, however, failures of platinum wires in the immersion thermocouple are almost invariably of the mechanical type, presumably owing to the relative softness of pure platinum wire.

Swelling of Wire

In all the oil tests in which the wire was inserted in a silica sheath, it was noted that the maximum attack was concentrated on the exposed wire just beyond the end of the insulator. It was also noticed that a marked swelling took place at this position, and that a maximum degree of hardness was recorded there (150 V.P.N.). A photograph ($\times$ 50) of the general appearance of the wire after such a test is shown in Fig. 7. The swelling will be noted. It would appear that as attack is concentrated at this spot, the swelling is a direct result of the intergranular penetration.

Spectrographic Examination of Platinum–13% Rhodium Thermocouple Wire

Through the courtesy of the Sheffield Smelting Co., Ltd., we report the results of their spectrographic examination of the two samples of wire referred to earlier.

"Two samples of wire, (*A*) partially annealed and (*B*) fully annealed, which had been subjected

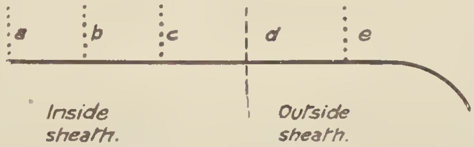


Fig. 2—Division of wire samples submitted for spectrographic examination

to the oil attack test of one drop of oil for 15 min. at 1400° C., were sent to us by Dr. Reeve for spectrographic examination, with particular reference to the area associated with a 'swelling' referred to in his report.

Each sample was divided into five areas as shown in the diagram Fig. 2, each area being examined separately. Area *d* is the area of

Table II—*Results of Spectrographic Examination*

Element	Before Test	After Test *a*	*b*	*c*	*d*	*e*
Sample A—Partially Annealed Wire						
Copper	v.m.t. 2	m.t. 1	m.t. 1	m.t. 2	f.t. 2	f.t. 2
Silicon	v.m.t. 1	v.m.t. 2	...	v.m.t. 2	f.t. 1	...
Magnesium	v.m.t. 1	v.m.t. 2	...	v.m.t. 2	m.t. 2	...
Nickel	Nil	Nil	Nil	v.m.t. 1	m.t. 1	v.m.t. 1
Iron	Nil	Nil	Nil	v.m.t. 1	v.m.t. 2	v.m.t. 2
Sample B—Fully Annealed Wire						
Copper	v.m.t. 2	m.t.2	f.t. 1	m.t. 2	f.t. 2	m.t. 1
Silicon	v.m.t. 1	v.m.t. 2	...	v.m.t. 2	m.t. 1	...
Magnesium	v.m.t. 1	v.m.t. 2	...	v.m.t. 2	m.t. 1	...
Nickel	Nil	v.m.t. 1	v.m.t. 1	v.m.t. 1	v.m.t. 2	v.m.t. 1
Iron	Nil	v.m.t. 1	v.m.t. 1	v.m.t. 1	v.m.t. 2	v.m.t. 1

Explanation of notation in order of increasing amount :
v.m.t. 1 = Very minute trace 1—least amount.
v.m.t. 2 = Very minute trace 2.
m.t. 1 = Minute trace 1.
m.t. 2 = Minute trace 2.
f.t. 1 = Faint trace 1.
f.t. 2 = Faint trace 2—greatest amount.

'swelling' and pronounced crystal-boundary etch.

The spectrographic report is attached hereto (in Table II).

The following features emerge from this report :

(i) The impurities picked up by the wire in the oil test are copper, silicon, magnesium, nickel, and iron, in decreasing order of amount.

(ii) The area of the 'swelling' with the pronounced crystal-boundary etch shows the greatest contamination. This is true of both samples.

(iii) In the case of the partially annealed wire, the exposed portion of the wire is more contaminated than the portion inside the silica insulator.

In the case of the fully annealed specimen, apart from the area of the 'swelling,' there is little to choose between the different portions of the wire, exposed or protected, from the point of view of greater contamination."

(End of report of Sheffield Smelting Co., Ltd.)

Summary and Conclusions

Fracture of wires used in the Schofield–Grace immersion thermocouple may be due to purely mechanical causes (such as damage to wire when passing over sharp edges of insulators) or to intercrystalline attack.

A test is described which simulates the intercrystalline attack on platinum and platinum–13% rhodium wires by heating a short length of wire in a graphite block, in the presence of a small quantity of oil, the wire being placed in a silica insulator to simulate the conditions in the actual thermocouple. Rapid attack takes place at temperatures above 1200° C. The test has been finally standardized at 1400° C. for 15 min., using one drop (0·05 c.c.) of lubricating oil Shell B. 6 of sulphur content 0·2%.

The results of this test on two batches of wires *A* (semi-annealed) and *B* (fully annealed), indicate that wire *A* is more prone to attack than wire *B*.

Grain-growth tests at elevated temperatures indicate that wire *A* coarsens more rapidly and gives a less homogeneous grain-size than wire *B*.

Similar tests carried out on a fully cold-worked wire, *C*, and a fully annealed wire *D* drawn from the same platinum–rhodium billet indicate that both wires are more resistant to oil attack than wire *A*. It is stated by the suppliers that the latter is only partially annealed, and its greater sensitivity to grain growth and intercrystalline attack is no doubt related to a critical degree of residual strain.

Attention is drawn to a peculiar swelling of wires used in the oil test, when the test is carried out in a silica insulator ; the swelling takes place at the portion of the wire just leaving the insulator. Micro-examination shows that the maximum concentration of intercrystalline attack takes place at this swelling.

Spectrographic examinations of wire *A* (partially annealed) and wire *B* (fully annealed) which have been subjected to the oil test, are reported with particular reference to the area associated with the swelling. It is shown that copper, silicon, manganese, nickel, and iron are present in decreasing order of amount, and that the greatest contamination takes place in the area of the swelling. This is true of both samples. The exposed portion of the wire, *i.e.*, beyond the insulator, is more contaminated in the case of the partially annealed than in the case of the fully annealed wire.

Another paper in this symposium, by Dr. Chaston and his co-workers, shows that both sulphur and silica play a part in the embrittlement which occurs, probably through the intermediary of volatile SiS_2. Modified oil tests are described which confirm these findings.

In practice it has been found that if the iron pipes used for carrying the thermocouple wires are thoroughly baked at a dull-red heat with a current of air drawn through them, almost complete freedom from failures of the intercrystalline type is obtained. It is important that this baking should be carried out after all pipe-fitting work on the tube is complete, and that no further oil should be used on any of the fittings once baking is complete.

Failures of the mechanical type due to damage of the wire by insulators, etc., can be prevented by using either glass tubing or woven-glass braid to carry the wires as far as possible, and by avoiding all sharp edges likely to get into contact with the wire.

Acknowledgments

The help of Mr. E. D. Harry and of Mr. H. K. M. Lloyd must be acknowledged in carrying out the tests described above.

The Management of the Appleby-Frodingham Steel Co., Ltd., must also be thanked for permission to publish the results of the investigation.

Section III—An X-Ray Investigation of the Embrittlement of Platinum and Platinum-Rhodium Wires*

By H. J. Goldschmidt, M.Sc., A.Inst.P.,† and T. Land, M.A., A.Inst.P.†

Synopsis

Platinum and platinum–rhodium thermocouple wires contaminated and embrittled in service or by experimental exposure to oil and graphite in the presence of silica, have been examined by the X-ray diffraction method.

Three phases additional to the matrix were found, two of which were identified as silicides. The more severe embrittlement of the platinum–rhodium wire is explained by the formation of a phase aggregate different from that in the platinum wire.

An additional observation on a platinum–rhodium wire at the point of brittle fracture in service is the segregation of a finely crystalline platinum-black type of phase free from rhodium, from the matrix which itself is rhodium enriched.

An incidental outcome of this work is the suggested determination of rhodium content in thermocouple wires by the X-ray diffraction method.

The embrittlement of rare-metal thermocouple wires is no new trial for the pyrometrist, but the use of rare-metal thermocouples in liquid-steel pyrometry has made such embrittlement a matter of serious financial consequence. It has been demonstrated by spectrographic methods that silicon reduced from siliceous refractory materials plays a major part in platinum contamination. The nature of the products of such a reaction can readily be demonstrated by the methods of X-ray crystallography, and it was thought worth while to use this technique to establish the identity of the constituents responsible for the embrittlement. In particular it has been noted that the rhodium-alloy wire is usually the more seriously embrittled, and some explanation of this fact was sought.

Standard Materials

Certain standard materials of known composition were first assembled. A series of wires were obtained having the following compositions :

(*a*) Pure platinum.
(*b*) Platinum–rhodium alloy containing $13 \cdot 02 \pm 0 \cdot 05$ wt.-% of rhodium.
(*c*) Platinum–rhodium alloy containing $20 \cdot 03 \pm 0 \cdot 05$ wt.-% of rhodium.
(*d*) Pure rhodium.

Platinum–rhodium alloys form a continuous series of solid solutions having a face-centred-cubic structure. The lattice parameters provide a convenient method of determining the rhodium content of any platinum–rhodium alloy, and this fact proved valuable in the investigation.

The next standard materials were prepared by adding 1% of silicon to small melts of pure platinum and platinum – 13% rhodium alloy. Filings from these synthetic alloys were prepared and gave X-ray pictures which contained, in addition to the platinum or platinum–rhodium lines, faint extra patterns due to silicides.

Finally, the following compounds were examined :

(*a*) Platinum oxide.
(*b*) Platinum sulphide.
(*c*) Rhodium oxide.
(*d*) Rhodium sulphide.

A sample of platinum black (colloidal platinum) was examined also.

Contaminated Wires

Furnished with these standard samples and their diffraction photographs, we examined six contaminated wires. These wires were prepared as follows :

The first pair of wires (*A* and *B*) were of normal thermocouple materials—one of pure platinum and the other of the 13% rhodium alloy. Two-inch lengths of each wire were sheathed in a similar length of fine twin-bore silica tubing, which was broken into short sections about ⅛ in. long. The sheathed wires were placed in a graphite tube, the inside of which was smeared with machine oil. The ends of the graphite tube were closed with asbestos wool, and the whole assembly was placed in a gas-fired furnace and maintained at 1300° C. for 3 hr. The procedure conforms in its essentials to the method proposed by Dr. Reeve in his paper.

The third wire (*C*) was a platinum–13% rhodium thermocouple wire which had accidentally become contaminated some months previously. It formed part of a thermocouple which was used to measure temperature changes

* Report submitted to the Liquid Steel Temperature Sub-Committee, 15th June, 1945.

† Research Department, Wm. Jessop & Sons, Ltd., Sheffield.

in a cylinder of steel during oil quenching from 850° C. The quenching oil had penetrated to the thermocouple, which failed at about 350° C. Only the platinum–rhodium wire was kept for examination, and this wire had fallen to pieces at the junction of the silica insulators. The contamination appeared to be localized at the point of fracture, and it was the fractured tip which was examined.

A pure rhodium wire was subjected to the silica, oil, and graphite treatment used in preparing samples A and B ; this contaminated wire is designated D. Finally, a thermocouple which had failed in use in a liquid-steel pyrometer was examined. The platinum wire is called E and the platinum–rhodium wire F.

Experimental Method

The wire specimens were examined in the 9-cm. dia. Debye-Scherrer camera, using cobalt and copper radiations, since for lattice-parameter measurements the former was most suitable at the platinum end, and the latter at the rhodium end of the series. Reflections (331) and (422)K_α formed convenient end doublets for the respective radiations. It should be mentioned that the X-ray analysis on the wires, owing to absorption, essentially reveals the state of the surface layers of the specimen. The platinum and rhodium oxides and sulphides were received as powders, and filings were taken from the silicon alloys ; these materials were also examined by the powder method.

The Platinum Rhodium System

Platinum-rhodium alloys form a continuous series of solid solutions of a face-centred-cubic structure. Figure 9 illustrates a series of platinum–rhodium solid solutions by their copper radiation photographs. Table III gives the lattice parameters (a_0) of pure platinum and rhodium as at present observed, in comparison with those found by Owen and Yates.[1] The agreement is good, but other rather diverging values are also given in the literature.

According to Weerts and Beck,[2] Vegard's law holds strictly between platinum and rhodium,

Table III—*Lattice Parameters of Pure Platinum and Pure Rhodium*

Authority	Lattice Parameter a_0, kX.	
	Platinum	Rhodium
Observed ...	3·9161 (± 0·0001)	3·7968 (± 0·0001)
Owen and Yates ...	3·9158 (± 0·0003)	3·7966 (± 0·0003)

i.e., the lattice parameters vary linearly with atomic composition. To check this statement, alloys of accurately known composition were examined and found to have the lattice parameters shown in Table IV.

Table IV—*Linear Variation of the Lattice Parameters of Platinum and Rhodium with Atomic Composition*

Rhodium, wt.-%	Rhodium, at.-%	Lattice Parameter a_0, kX.
0	0	3·9161 (± 0·0001)
13·02 (± 0·05)	22·12	3·8908 (± 0·0002)
20·03 (± 0·05)	32·21	3·8788 (± 0·0002)
100	100	3·7968 (± 0·0001)

In Fig. 3 these values are plotted against the atomic percentage of rhodium, and it will be seen that there is a small deviation from the linear relationship. Such small deviations from Vegard's law are quite normal in alloy systems. The validity of the curve at the rhodium-rich end can, of course, only be presumed, but for the present purpose only the range between 0 and,

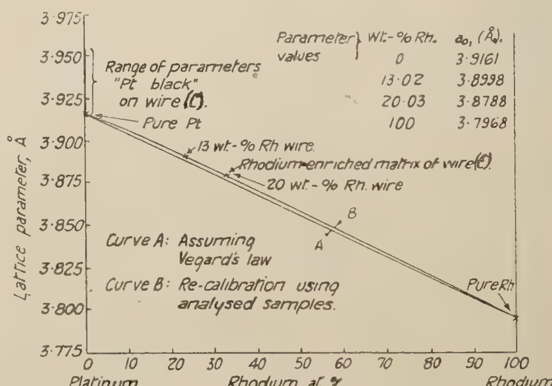


Fig. 3—Lattice parameters of platinum–rhodium alloys

say, 30% of rhodium is of interest. Within this range it is possible to determine the rhodium content from the measured lattice parameter to about ± 0·1%, which approaches the accuracy of determinations by chemical analysis. The atomic percentage of rhodium may be calculated from the lattice parameter, using the following equation :

$$c = 903 \cdot 1\,(3 \cdot 9161 - a_0) - 1296\,(3 \cdot 9161 - a_0)^2 + 6300\,(3 \cdot 9161 - a_0)^3,$$

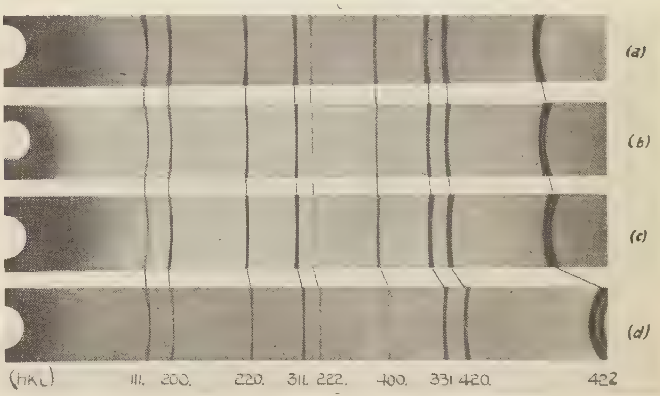
where c is the composition in atomic per cent. of rhodium.

The possible occurrence of a superlattice of the type Pt_3Rh in the platinum–rhodium system had to be taken into account, although it has never been reported. However, no superlattice

FIG. 4—Intercrystalline penetration near fracture of platinum-rhodium wire

FIG. 8—Wire *B* after oil test, using one drop at 1,400° C. × 50

FIG. 5—Wire *A* after oil test, using one drop at 1,400° C. × 150



(*a*) Pure Pt (*c*) Pt–20% Rh
(*b*) Pt–13% Rh (*d*) Pure Rh

FIG. 9—X-ray photographs of a series of platinum–rhodium solid solutions, with CuK radiation (Kα and Kβ)

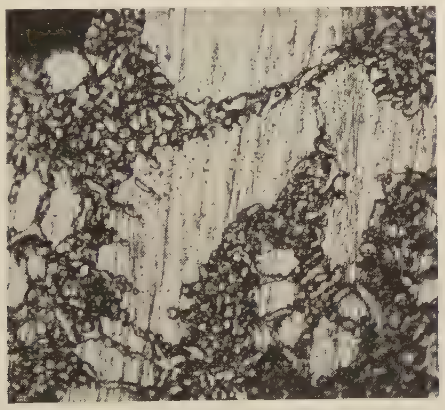


FIG. 6—Wire *A* after oil test. Central section of wire × 406

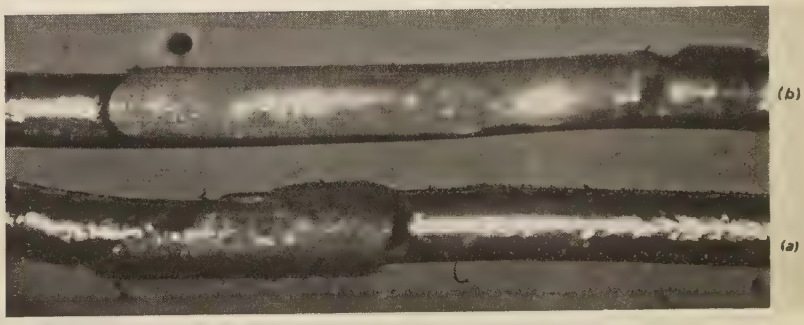


Fig. 10—Appearance of embrittled wires (× 25 approx.)
(*a*) Pt wire (*b*) Pt–13% Rh wire

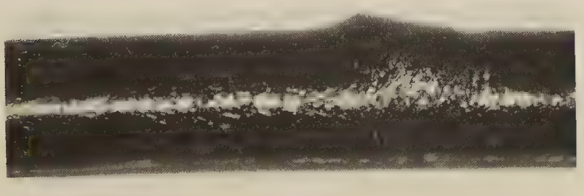


FIG. 7—Swelling of wire after oil test (reduced to four-fifths linear in reproduction) × 50

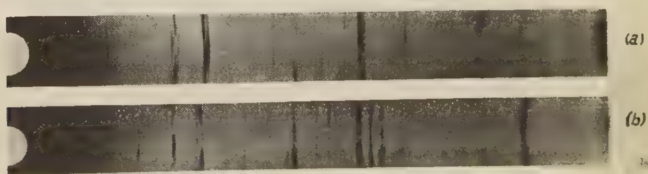


FIG. 11—X-ray photographs of embrittled wires *A* and *B* (CoKα radiation) showing silicides. (*a*) Pt wire (*b*) Pt–13% Rh wire

Figs. 4 to 6, and 8 reduced to three-quarters linear in reproduction

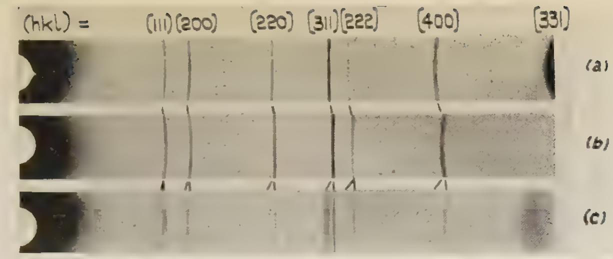


FIG. 12—X-ray photographs of embrittled wire C and pure platinum and platinum–13% rhodium wires. (CoKα radiation.) (*a*) Pure Pt wires (*b*) Pt–13% Rh wire (*c*) Contaminated Pt–13% Rh wire

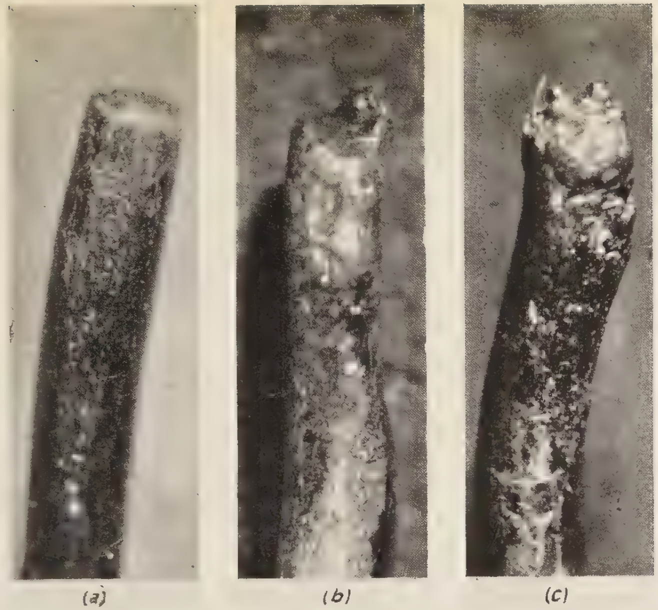


FIG. 13—Photographs showing the rounded appearance of the fractured ends of the wires, indicating that melt had been present. (*a*) Couple *A*, Pt–Rh wire (*b*) Couple *B*, Pt wire (*c*) Couple *B*, Pt–Rh wire

FIG. 14—Fractured end of a wire from thermocouple *C*, indicating hot tensile conditions in addition to showing the rounded appearance as in Fig. 13

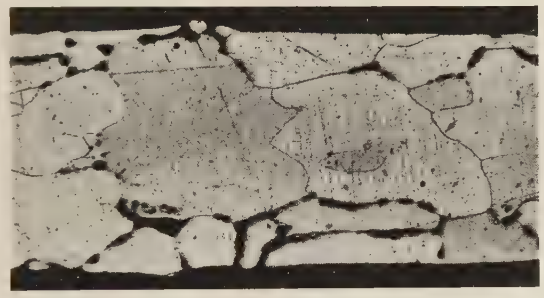


FIG. 15—Microstructure of platinum wire heated at 1400° C. for 1 hr. with sulphur in a graphite block

FIG. 16—Cross-section of pure platinum wire at point of failure. Unetched × 100

FIG. 17—Cross-section of pure platinum wire at point of failure. Etched ×100

FIG. 18—Cross-section of pure platinum wire at point of failure. Etched ×180

Figs 15 to 18 reduced to three-quarters linear in reproduction

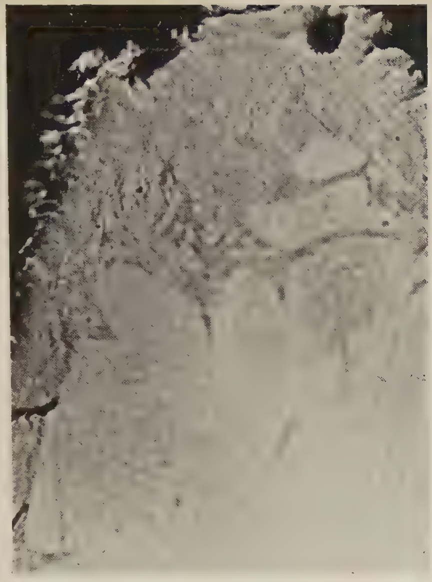


FIG. 19—Cross-section of platinum–13% rhodium wire at point of failure. Unetched × 375

FIG. 20—Cross-section of platinum–13% rhodium wire at point of failure. Etched × 180

FIG. 21—Pure platinum wire of hot junction of top thermocouple. Etched × 100

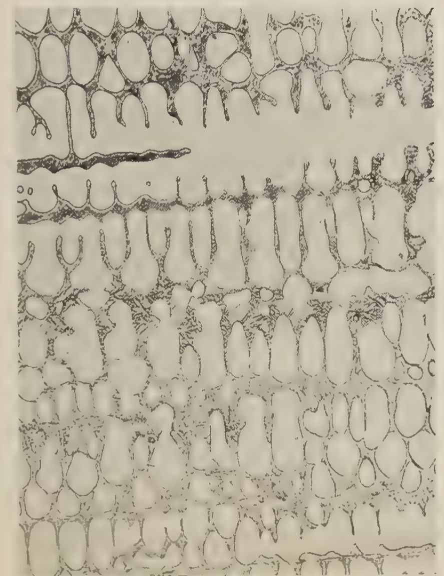


FIG. 22—Pure platinum alloyed with 1% of silicon. Etched × 180

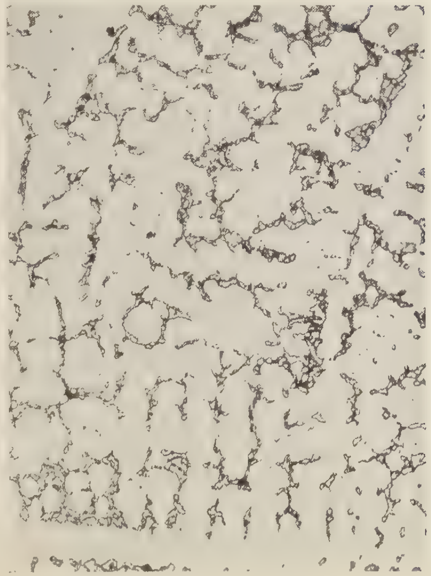


FIG. 23—Platinum–13% rhodium alloyed with 1% of silicon. Etched × 180

FIG. 24—Platinum–13% rhodium wire at point of fracture showing intercrystalline attack. Unetched × 180

Figs. 19 to 24 reduced to four-fifths linear in reproduction

FIG. 25—Platinum–13% rhodium wire at point of fracture, showing intercrystalline attack. Etched × 180

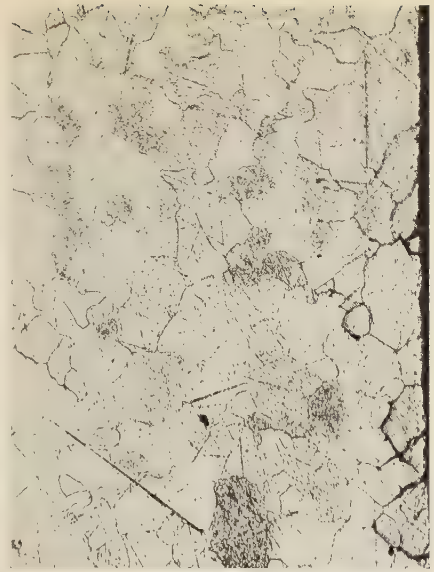


FIG. 26—Microsection of platinum–13% rhodium wire 2 mm. from point of fracture. Etched × 180

FIG. 27—Platinum–13% rhodium wire about to break, showing intercrystalline cracks reaching nearly across the section of the wire. Etched ×180

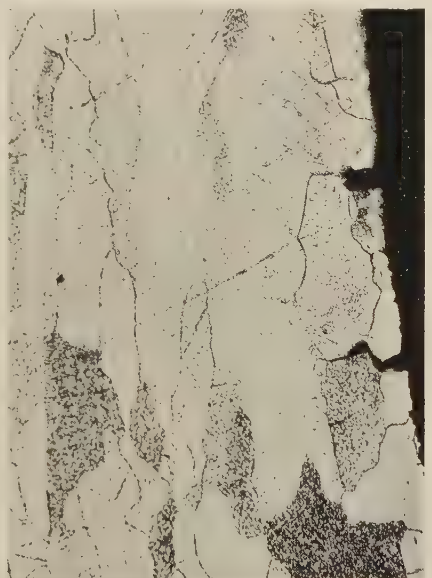


FIG. 28—Pure platinum wire showing intercrystalline cracks to surface layer. Etched ×180

FIG. 29—Cross-section through contaminated end of platinum–13% rhodium wire showing contaminant at the grain boundaries. Etched × 180

FIG. 30—Cross-section through contaminated end of platinum–13% rhodium wire showing contaminant at the grain boundary. Etched × 375

Figs. 25 to 30 reduced to four-fifths linear in reproduction

lines were found in the positions to be expected theoretically.

Platinum–Rhodium Silicides

X-ray photographs taken of filings from the small platinum and platinum–rhodium ingots to which silicon had been added, show the normal platinum and platinum–rhodium lines, and also faint patterns additional to the main phase. It was concluded that these correspond to a platinum silicide in the case of the platinum melt, and another platinum or mixed platinum–rhodium silicide in the platinum–rhodium melt. The main point is that the two silicides differ entirely from one another. To decide the actual composition of the silicides would require a complete survey of the platinum–rhodium–silicon ternary system which is quite outside the scope of the present investigation. In the platinum–silicon binary system, the compounds Pt_5Si_2, Pt_2Si, Pt_3Si_2, Pt_4Si_3, PtSi, and $PtSi_{12}$ have been variously reported (Colson,[3] Lebeau and Novitzky,[4] and Baraduc-Muller[5]). The only recent work on the system is that by Voronov,[6] who identified the embrittling compounds Pt_5Si_2, Pt_2Si, and PtSi formed when platinum apparatus is heated in contact with siliceous materials under reducing atmospheres.*

An X-ray study of the platinum–silicon system does not seem to have been undertaken, and would provide useful information. No information at all is available regarding rhodium–silicon alloys.

Since the present silicides are obviously in co-existence, if not in equilibrium with the platinum matrix, it is highly probable that they correspond to the platinum-richest ones, *viz.*, one to Pt_5Si_2 and the other to Pt_2Si, or to a rhodium or mixed platinum–rhodium silicide.

The possibility of platinum or rhodium silicates forming has been considered, but may be discarded. The existence of such silicates has never been reported in the literature. Mellor[7] does not mention them, while he discusses the platinum silicides at length as readily formed. Oxygen when present reacts directly with silicon to form SiO_2. In corroboration it was noted that an X-ray photograph of filings from the top surface of the platinum + 1% silicon ingot reveals the presence of a small amount of quartz.

The lattice parameter of the platinum matrix in this alloy has been measured to be 3·9146 kX. compared with the value of 3·9161 kX. for pure platinum. This is interesting as it shows:

(i) That a small degree of solubility of silicon in platinum exists.

(ii) That silicon goes into solution in the platinum lattice not interstitially, but by substitution of metal atoms. Only such substitution would account for the observed contraction, as the silicon atom is of smaller size (atomic radius of Pt is 1·38 kX.; of Si, 1·17 kX.).

Summarizing, we may then say that silicon has a small solubility in platinum, and forms a number of silicides, of which two have been detected. One formed by the addition of 1% of silicon to the molten platinum will be designated silicide "*P*," and the other formed by adding 1% of silicon to the 13% rhodium alloy will be called silicide "*R*." The exact compositions of the two silicides are uncertain.

Platinum and Rhodium Compounds

The photographs of the platinum and rhodium oxides and sulphides showed poor crystallinity, but were good enough for it to be possible to say that none of the compounds analysed appeared in any of the contaminated wires. The platinum black gave a pattern identical in mean spacing with the pure platinum wire, but the lines showed marked broadening owing to the small particle size.

Examination of Contaminated Wires

Contaminated Wire A

This was a pure platinum wire, contaminated by the silicon, oil, and graphite method. Its

Table V—*Interplanar Spacings of Embrittled Wire* A

The figures in italics correspond to the platinum lines

Platinum Wire A		Platinum + 1% Silicon Alloy	
Spacing, kX.	Intensity	Spacing, kX.	Intensity
3·9	m	3·9	vw
3·2	m	3·2	vw
2·76	w	*2·76*	vw
2·23	s	2·24	s
2·10	w	2·10	w
1·94	s	*1·95*	s
1·74	m	1·74	vw
1·49	m	1·50	vw
1·385	s	*1·385*	s
1·315	m	1·315	w
1·228	m	1·230	w
1·181	s	*1·182*	s
1·130	m	*1·132*	m
1·095	m	1·100	vw

s = Strong, m = Medium, mw = Medium to weak, w = Weak, vw = Very weak

* The compound $PtCu_2Si$ has been reported as very stable, and readily formed in the presence of traces of copper. This is interesting as spectrographic analysis frequently shows the presence of copper in contaminated platinum wire.

surface had a scale-like appearance, with irregular swellings protruding at points corresponding to the gaps between the short lengths of silica insulator. The appearance of the wire is illustrated in Fig. 10. The interplanar spacings are shown in Table V, together with the corresponding spacings found in the synthetic platinum–silicon alloy. It will be seen that the agreement between the two sets of lines is extremely good, and there can be no doubt that the same silicide is present in the embrittled wire as in the synthetic alloy. The X-ray photograph is shown in Fig. 11. Since the silicide was concentrated at the surface of the wire, its diffraction pattern is very prominent, showing lines of strength comparable with those of the matrix. Furthermore, considerable crystal growth has occurred due to heating at 1300° C. for 3 hr. This is manifested by the discontinuous (spotty) lines. The lattice parameter of the contaminated wire was 3·9156 ± 0·0002 kX., indicating a slight solid solution of the silicon, but not as extensive as in the synthetic alloy. The non-uniformity of lines reduces the accuracy of measurement in this case, and the observed difference is only just outside the limit of error.

Contaminated Wire B

This wire, also shown in Fig. 10, was a platinum–rhodium wire contaminated in the same way as wire *A*, and had the same scaly appearance. The interplanar spacings are shown in Table VI, in which it is compared with the synthetic platinum–rhodium + 1% silicon alloy. Once again the

TABLE VI—*Interplanar Spacings of Embrittled Wire* B

The figures in italics correspond to the platinum-rhodium lines

Platinum–Rhodium Wire *B*		Platinum–Rhodium + 1% Silicon Alloy	
Spacing, kX.	Intensity	Spacing, kX.	Intensity
2·80	m	2·80	w
2·32	m	2·32	w
2·23	s	*2·23*	s
1·98	m	1·99	vw
1·94	s	*1·94*	s
1·61	m	1·61	vw
1·41	m	1·41	vw
1·385	s	*1·385*	s
1·259	m	1·258	vw
1·172	s	*1·172*	s
1·148	s	1·148	w
1·122	s	*1·122*	s
0·995	m	0·995	w

s = Strong, m = Medium, mw = Medium to weak, w = Weak, vw = Very weak

agreement is within the limits of experimental error, and there is no room for doubt that the second silicide (silicide "*R*") is present in both specimens. The X-ray photograph is shown in Fig. 11. Again the silicide was concentrated at the surface, and the crystallinity is very coarse.

Contaminated Wire C

This wire was contaminated in very different circumstances from the first two, and the products of the reaction are very different. The original wire was a 13% rhodium alloy. Four different phases can be detected from the X-ray photograph, which is reproduced in Fig. 12. In the first place the original single-phase platinum–rhodium alloy has split into two face-centred-cubic phases, one of which (the matrix) is enriched in rhodium and gives a sharp pattern. The other phase, enriched in platinum, produces a highly diffuse pattern of parameters greater than that of platinum.* Spacing measurements show that the matrix has a lattice parameter of 3·8795 kX. corresponding to 19·6 wt.-% of rhodium, while the lattice parameter for the dispersed phase ranges from approximately 3·92 to 3·965 kX. These values are shown in the diagram, Fig. 3.

The fact that the dispersed phase thus shows lattice dimensions larger than those of pure platinum can well be accounted for by an interstitial solid solution of certain foreign atoms in platinum, expanding the lattice. The line broadening is explained by a heterogeneous series of solid solutions, covering a range of compositions. However, in addition it is highly probable that the diffuse pattern is due to small particle size, the effect of which is superimposed on that of composition variations.

If the line broadening were due to particle size alone, a calculation from the line breadth gives a particle size of approximately 100 Å. This is in good agreement with the order of magnitude reported for platinum black, and found experimentally in the platinum black which we investigated. The actual particle size will be somewhat larger, as the heterogeneity effect is certainly contributory. This is indicated by the intensity distribution across each line complex, which should be approximately gaussian for the effect of small particle size alone, but is irregular. The fact that the lattice parameters for this presumed platinum-black range upwards from the platinum value, also shows the presence of foreign elements causing heterogeneity in composition.

* It should be noted that the slightly eccentric line positions of the platinum-black pattern in Fig. 12 are due to the deposit being concentrated at the wire tip only.

Colloidal platinum is known readily to take into solution impurities such as carbon, sulphur, and oxygen or hydrogen, adsorbed from the system in which it was produced. Entering interstitially, these would all expand the lattice, except perhaps hydrogen, which has been reported to contract the lattice.[8] Some relevant references to this subject are Thomson, Finch, and others,[9] Richards [10] (platinized asbestos), and Uspensky and Konobejewsky.[11]

Faint extra lines appear in addition to the principal ones. Comparison with the synthetic silicon alloys shows the presence of the platinum

Table VII—*Interplanar Spacings of Embrittled Wire* C

The figures in italics correspond to the platinum or platinum-rhodium lines

Platinum–Rhodium Wire *C*		Platinum + 1% Silicon Alloy	
Spacing, kX.	Intensity	Spacing, kX.	Intensity
3·9	vw	3·9	vw
3·2	vw	3·2	vw
2·97	mw		
2·76	w	2·76	vw
2·45	vw		
2·22	s	*2·24*	s
2·10	vw	2·10	w
1·93	s	*1·95*	s
1·86	vw		
1·75	vw	1·74	vw
1·67	vw		
1·60	vw		
1·52	vw	1·50	vw
1·44	vw		
1·375	s	*1·388*	s
1·353	w		
1·32	vw	1·315	w
1·235	vw	1·230	w
1·172	s	*1·182*	s
1·122	s	*1·132*	s
1·100	vw	1·100	vw

s = Strong, m = Medium, mw = Medium to weak, w = Weak, vw = Very weak

silicide which we have called silicide "*P*." This is interesting, since the contaminated platinum–rhodium wire *B* contained not this silicide "*P*," but the silicide "*R*," found in the synthetic platinum–rhodium–silicon alloy.

The silicide "*P*" accounts for most of the faint lines on the X-ray picture of this contaminated wire but a few other lines remain to be accounted for, as is seen in Table VII, which shows the interplanar spacings (omitting the platinum-black reflections).

Comparison with the oxides and sulphides of platinum and rhodium, and with numerous other compounds, has failed to identify the additional phase which we call "*Q*." It was thought that it might possibly be a silicide of rhodium, and a pure rhodium wire (*D*) was therefore contaminated by the usual silica, oil, and carbon method. Although numerous extra lines occurred on the X-ray photograph of the contaminated rhodium wire, there was no correspondence between the contaminated products of the pure rhodium wire and those of the platinum–rhodium wire *C*. It is not unlikely that the unidentified constituent "*Q*" is a further platinum or platinum–rhodium silicide.

Summarizing, the constitution of wire *C* is:

(i) Platinum–rhodium solid solution with 19·6 wt.-% of rhodium as against 13% of rhodium before contamination.

(ii) Finely dispersed platinum (platinum black), with interstitial lattice impurities, probably carbon, sulphur, or oxygen.

(iii) Platinum silicide of the type "*P*" found in the synthetic platinum–silicon alloy.

(iv) A trace of an additional phase, "*Q*."

Contaminated Wires E *and* F

These wires formed part of a thermocouple which failed in service in a liquid-steel pyrometer. The rhodium-alloy wire was broken when the pyrometer was examined, and the X-ray examination was made at a point close to the fracture. The X-ray photographs showed that the platinum wire had traces of both the platinum silicide "*P*" and the unidentified phase "*Q*," while the platinum–rhodium wire had traces of the phase "*Q*" only. The interplanar spacings of the extra phases in wires *E* and *F* are set out in Table VIII.

Table VIII—*Interplanar Spacings of Wires* E *and* F

All intensities very weak

Platinum Wire *E*	Platinum–Rhodium Wire *F*
Spacing, kX.	Spacing, kX.
2·95	2·95
2·76	
2·45	2·45
2·10	
1·87	1·86
	1·68
1·64	1·60
1·52	1·45
	1·35
1·31	1·30
1·23	
1·095	1·085
0·991	
0·967	

Discussion

The contamination of the various wires produced a number of new phases which are set out in Tables IX and X.

In addition to the new phases set out in Tables IX and X, several other effects were noticed. Of particular interest is the effect found in the platinum–rhodium wire *C* in which the 13% rhodium matrix split into two phases, one containing nearly 20% of rhodium and the other containing no rhodium. There is clear evidence in this case that an element other than silicon has also reacted with the platinum. This element enters interstitially into solid solution with the pure platinum, but not with the rhodium alloy. Other contaminated samples show that silicon enters into solid solution to a small degree with both platinum and platinum–rhodium.

The investigation provides a possible explanation of the greater brittleness of contaminated platinum–rhodium wires in comparison with contaminated platinum wires. All the contaminated platinum specimens examined contained the silicide "*P*," and none contained the silicide "*R*." Of the contaminated platinum–rhodium samples, two contained only silicide "*R*" and the other two contained the phase "*Q*," in one case alone and in the other case in conjunction with the silicide "*P*." It would appear that silicide "*P*" is relatively strong and ductile, whereas silicide "*R*" and the phase "*Q*" would appear to be weak and brittle. It is true that both phases "*P*" and "*Q*" occur in conjunction in both the brittle platinum–rhodium wire *C*, and the undamaged platinum wire *E*, but the difference may well be due to the manner of distribution of the two phases. In wire *E* very little of phase "*Q*" was present, and its embrittling effect may have been neutralized by its crystallites being totally surrounded by the matrix and the silicide "*P*." Furthermore, the wire *C* is exceptional in that the matrix was split into the two phases, one of which contained no rhodium. Tables IX and X suggest that silicide "*P*" is always associated with a pure platinum matrix, and it is quite possible that in wire *C*, phase "*Q*" is confined to the rhodium-rich part of the matrix and silicide "*P*" to the platinum-black portion.

Table IX—*Summary of Contamination Products—Platinum*

Specimen	New Phases
Platinum + 1% silicon alloy ...	Silicide "*P*."
Platinum wire *A* heated with oil and graphite	Silicide "*P*."
Platinum wire *E* from liquid-steel pyrometer	Silicide "*P*" and phase "*Q*."

Table X—*Summary of Contamination Products—Platinum–Rhodium*

Specimen	New Phases
Platinum–rhodium + 1% silicon alloy.	Silicide "*R*."
Platinum–rhodium wire *B* heated with oil and graphite.	Silicide "*R*."
Platinum–rhodium wire *C* from couple accidentally contaminated by oil.	Silicide "*P*" and phase "*Q*." Matrix split into two phases, one free from rhodium (platinum black), the other containing 19·6% of rhodium.
Platinum–rhodium wire *F* from liquid-steel pyrometer.	Phase "*Q*."

Acknowledgments

The authors wish to record their thanks to Mr. D. A. Oliver, Director of Research, Wm. Jessop & Sons, Ltd., for his advice and encouragement. They are greatly indebted to Mr. R. C. Jewell and the Sheffield Smelting Co., Ltd., for the provision of platinum and rhodium compounds, and synthetic alloys of platinum and silicon, and platinum–rhodium and silicon; also to Dr. J. C. Chaston and Messrs. Johnson, Matthey & Co., Ltd., for the analysed series of platinum–rhodium alloys.

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Section IV—Contamination and Failure of Rare-Metal Thermocouples*

By D. Manterfield, F.I.M.†

Synopsis

During an investigation into the solidification and cooling of steel ingots, failure of thermocouples occurred due to serious contamination of the couple wires. Subsequent visual, X-ray, microscopic, and spectrographic examination showed this failure to be due to the reduction of silica to silicon and alloying of this silicon with platinum to form a new phase of platinum silicide.

Experimental

Unusual cases of bad contamination and failure of thermocouples have recently been experienced during an investigation into the thermal history of ingots during and after solidification.

Three thermocouples were arranged at different depths (1 ft. 6 in., 3 ft., and 4 ft. 6 in. from the bottom) in the centre of two different ingot moulds before teeming. The individual couples were insulated by twin-bore silica insulators and the triple assembly enclosed in a closed-end silica tube which extended above the ingot top. From the top of this silica tube to the potentiometer the individual wires were protected by fireclay beads.

Temperature measurements were taken at frequent intervals during casting and continued during cooling and solidification of the ingots. About 20 min. after casting all six couples failed almost simultaneously, the temperatures then being about 1480° C.

On examination the portions of wire above the point of fracture could be withdrawn, and it was found that all of the twelve thermocouple wires (six pairs) had failed by fracture at a point about 2 ft. 6 in. from the ingot top. On sawing the ingots into blocks the remainder of the couple wires were recovered and all were examined.

Visual examination revealed that all of the couples had fractured by melting at approximately the same position. The surfaces of the wires at this point were roughened and the fractured ends rounded. One individual couple had a fine, white, powdery deposit on the wire surface. The silica insulators at this position were coated for an inch or two with a black deposit which appeared to be of a tarry nature. The nature of this deposit was not definitely established, but it was apparently carbonaceous. Some of the silica insulators at the point of failure were also devitrified and had a deposit of white powder on them. The wires at the bottom, intermediate, and extreme top (*i.e.*, well below and above the failure point) were in perfect condition.

The upper portions of three thermocouples were submitted to our Research and Development Department for examination and calibration, and three were sent to The Sheffield Smelting Co., Ltd., for micro- and spectrographic examination. Subsequently, further samples of uncontaminated couples were also sent to The Sheffield Smelting Co., Ltd.

Reports of The United Steel Companies, Ltd. (*Research and Development Department*)

Extracts are here given from the three reports on the thermocouple wires submitted.

(i) *Visual Examination*

The fractured ends of the wires all had a rounded appearance indicating that local fusion had occurred due to the melting point of the wires having been considerably lowered by contamination.

Certain wires appeared to have been subjected to hot-tensile conditions.

Photographs (Figs. 13 and 14) illustrate these conditions.

(ii) *Palladium-Point Calibration*

Palladium-point calibration (1555° C.) was carried out on two thermocouples, the platinum and platinum–rhodium wires being connected at their fractured ends.

Thermocouple *B* ...	13° C. low at 1555° C.
Thermocouple *C* ...	7° C. low at 1555° C.

These errors were not outstandingly large. If, however, the contamination had been extremely local, the maximum e.m.f. might have been at a point away from the junction, in which case the above figures would not necessarily have been a measure of the degree of contamination at the actual hot junction.

(iii) *X-Ray Examination*

The third thermocouple was passed on to the X-ray section for examination.

* Report presented to the Liquid Steel Temperature Sub-Committee, 19th June, 1945.

† Messrs. Steel Peech & Tozer, Sheffield.

The wire samples were examined by the use of a 9-cm. dia. powder camera. Platinum–silicon (1% of silicon) and platinum–rhodium–silicon (1% of silicon) alloys supplied by The Sheffield Smelting Co., Ltd., were used as reference standards for the identification of the silicon compounds.

The results of the X-ray examination are given in Table XI.

Comments

(1) The silicon compounds are found over a distance of ¾-in. from the failure point, and increase in amount up to the failure point.

(2) The presence of a silicon-rich phase, especially on the platinum–rhodium wire, is probably caused by incomplete alloying of the silicon and the wire. It is probable that this silicon phase is confined mainly to the surface layers.

TABLE XI—*Results of X-Ray Examination*

Sample Position	Phases	Amount	Spacing, kX.
	(a) Platinum Wire.		
1/32 in. from failure point	Platinum rich	Large	3·913
	Platinum–silicon compound	Medium to small	—
	γ–iron	Very small	3·60
	α–iron	Very small	2·865
	Magnesium silicate	Small	—
	Magnesium hydroxide	Small	—
3/16 in. from failure point	Platinum rich	Large	3·913
	Platinum–silicon compound	Small	—
	Silicon rich	Trace	5·43
	Magnesium silicate	Very small	—
	Magnesium hydroxide	Very small	—
7/16 in. from failure point	Platinum rich	Large	3·910
	Platinum–silicon compound	Very small	—
	γ–iron	Very small	3·60
9/16 in. from failure point	Platinum rich	Large	3·911
	Platinum–silicon compound	Very Small	—
	γ–iron	Trace	—
	Other phases not identified	Very small	—
¾ in. from failure point	Platinum rich	Large	3·910
	Platinum–silicon compound	Trace	—
	γ–iron	Trace	—
	Other phases not identified	Very small	—
2 in. from cold end (after softening by electric heating)	Platinum rich	Large	3·912
	Platinum–silicon compound	Trace	—
	γ–iron	Very small	3·60
	Other phases not identified	Small	—
	(b) Platinum–Rhodium Wire		
1/32 in. from failure point	Platinum–rhodium rich	Large	3·889
	Platinum–rhodium–silicon compound	Small	—
	γ–iron	Small	3·60
	Silicon rich	Very small	5·44
7/16 in. from failure point	Platinum–rhodium rich	Large	3–888
	Platinum–rhodium–silicon compound	Very small	—
	γ–iron	Very small	—
	Silicon rich	Small	5·44
2 in. from cold end (after softening by electric heating)	Platinum–rhodium rich	Large	3·889
	Platinum–rhodium–silicon compound	Trace	—
	γ–iron	Very small	—
	Silicon rich	Medium to small	5·43

(3) The iron-rich phases presumably indicate surface contamination by fumes.

(4)—(*a*) The presence of magnesium silicate is caused either by :

(i) Oxidation of surface layers, and subsequent reaction of magnesia and silica or,

(ii) deposition of magnesium silicate from fumes.

(*b*) The presence of magnesium hydroxide is presumably due to hydration of some originally uncombined magnesia.

These magnesium compounds are probably confined to the surface layers.

(iv) *Examination by The Sheffield Smelting Co., Ltd.*

The following salient points are evident from Mr. Jewell's report, full details of which are reported in his contribution.

The microstructure showed clearly that the wire had been attacked by silicon forming a new phase of platinum silicide. Both the pure platinum and the platinum–13% rhodium wire were similarly attacked, but the attack was more pronounced in the case of pure platinum.

Spectrographic examination of the fractured ends of one couple confirmed the presence of silicon as the major contaminant ; with iron, copper, and magnesium as impurities in decreasing order of importance.

From the evidence of the microstructure, failure of the thermocouples was caused by the alloying of silicon with platinum, forming a fusible phase. The silicon was derived from the fused-silica sheath, the silica being reduced to silicon by the hydrogen or carbon monoxide gases given off by the steel as it solidified. The contraction of the steel on cooling probably cracked the outer sheath, thus permitting the diffusion of the gases to the inner sheath.

Section V—Embrittlement of Platinum/Platinum-Rhodium Thermocouples*

By J. C. Chaston, Ph.D., B.Sc., A.R.S.M.,† R. A. Edwards,† and F. M. Lever, B.Sc., A.R.C.S., A.R.I.C.†

SYNOPSIS

The embrittlement of platinum thermocouple wires heated in various environments has been studied. Platinum is not embrittled by heating in contact with carbon, hydrocarbon vapours, or sulphur alone. It is shown, however, that if platinum is heated in (though not necessarily in contact with) a silicious refractory enclosure in the presence of small amounts of sulphur and carbon (as is provided, for instance, by traces of oil), embrittlement may occur. It is suggested that in these conditions the volatile compound SiS_2 may be formed and that this attacks the platinum and forms platinum silicides at the crystal boundaries.

INTRODUCTION

Many users of immersion platinum/platinum–rhodium thermocouples have observed that the wires frequently become embrittled during use. Embrittlement generally occurs at a short distance from the hot junction and is reported to be found more frequently in the platinum–rhodium than in the pure platinum wires.

In a report by Mr. Land (in this symposium), it is shown that brittle fractures are associated with the use of new batches of steel tubing in the pyrometer-head assembly, and that by ' burning out ' the tubes before use (by heating and then blowing air through the tubes intermittently over a period of 1 hr.) embrittlement of the platinum and platinum-alloy wires can be prevented. It is concluded that the embrittlement seems to be caused by foreign matter in the steel tube. Most probably oil and other carbonaceous materials are responsible.

In these experiments it was found that the wires were not embrittled by heating in contact with graphite, and a similar observation is recorded in a more recent report to the Sub-Committee by L. Reeve (in this symposium). Reeve, again in substantial agreement with Land, found that embrittlement could, however, be produced in the presence of oil, and developed a test in which short lengths of wire were heated to 1400° C. for 15 min. with 0·05 c.c. of Shell B.6 heavy lubricating oil in a hole drilled in a graphite block. By this means, differences in the

* MS received by the Liquid Steel Temperature Sub-Committee, 7th July, 1944.

† Research Laboratories, Johnson, Matthey & Co., Ltd., Wembley, Middlesex.

susceptibility to attack of various batches of wires were compared.

While these experiments suggest very strongly that the presence of oil plays an important, if not essential, part in the embrittlement of thermocouple wires, the mechanism of the action has remained obscure. It has been felt that it would help in avoiding unsuitable conditions of service if the causes of embrittlement were more fully understood, and for this reason the effects have been studied in rather more detail.

Experimental

Samples of pure platinum and of platinum–13% rhodium alloy in the form of wires 0·020 in. in diameter were heated in a tube furnace arranged so that the atmosphere could be kept under control. In most of the experiments the wires were arranged horizontally to bridge a gap hollowed out in a small block of graphite or zircon, so that only the extreme ends of the wires were in contact with the support. In a few experiments the wires were placed in a cavity drilled in a graphite block, as used by Reeve. Unless otherwise stated, the furnace tube was of Morgan's 'Triangle H' material, an impervious, aluminous, porcelain refractory.

Carbon

Samples heated at 1400° C. for $\frac{1}{4}$ hr. in powdered graphite showed no signs of attack, thus confirming the observations by Land and by Reeve. Following a suggestion that hydrocarbons might cause embrittlement if allowed to crack on a platinum surface, samples were heated on a graphite support for periods of up to 1 hr. at 1200° C. in a current of hydrogen which had been passed through

(*a*) Xylol, or
(*b*) White Spirit.

Heavy deposits of carbon were found in the tube, particularly when using the vapour from white spirit, but no signs were found of any attack on the platinum or platinum–rhodium wires, and they remained ductile.

These results were not entirely unexpected. It is well established that if platinum is melted in graphite, the molten metal dissolves carbon which, on cooling, separates out as graphite and gives rise to great brittleness. The solubility of carbon in solid platinum is thus very small, and it seems unlikely that carbon will diffuse readily into platinum at temperatures below the melting point.

The results also suggest that the embrittlement of platinum wires when heated in the presence of oil may be due to some other cause than the presence of hydrocarbons. This conclusion was strengthened by the results of tests in which wires were heated at 1400° C. for 1 hr. in a cavity in a graphite block with pure liquid paraffin. No embrittlement was detected and the microstructure of the wires remained normal, even when as much as 1 c.c. of liquid paraffin was used. A similar treatment, using four drops of Mobiloil B.B. oil, resulted in severe embrittlement.

Sulphur

It is understood that Shell B.6 oil contains about 0·2% of sulphur. The effect of this element alone was therefore studied by passing sulphuretted hydrogen, sulphur dioxide, and mixtures of hydrogen and sulphuretted hydrogen over samples supported on a zircon block and heated at 1200° C. for $\frac{1}{2}$ hr. No traces of embrittlement were observed after any of the treatments.

Sulphur plus Carbon

When, however, both sulphur and carbon were present, severe embrittlement was produced in samples heated in a tube of 'Triangle H' material in a wire-wound electric-resistance furnace. In preliminary tests, wires were heated at 1400° C. for 1 hr. in a cavity in a graphite block with 0·35 g. of sulphur, and for $\frac{1}{2}$ hr. with 0·2 g. of sulphur. After these treatments, the wires fell to pieces if an attempt were made to bend them, and micro-examination showed the presence of large amounts of an intercrystalline constituent as shown in Fig. 15. Similar attack occurred on wires heated at 1200° C. for $\frac{1}{2}$ hr. in hydrogen or nitrogen which was bubbled through carbon disulphide. No attack was observed, however, at 1000° C. or at 1100° C. in these conditions.

No simple explanation for these results was apparent, and a careful chemical analysis was therefore made of a quantity of fine platinum wires which had been embrittled by heating (supported at their ends on a graphite block) at 1200° C. for 1 hr. in a mixture of nitrogen and carbon disulphide. The results were unexpected, since no trace of carbon or sulphur could be detected in the brittle wire which contained, however, 1·1% of silicon.

A search of the literature for a possible explanation of this result revealed that when silica or silicates are heated with carbon and sulphur a volatile silicon sulphide, SiS_2, may be formed.* This compound, it has been stated, is not readily formed in the absence of carbon, the action of which, whether derived from oil or any other carbonaceous material, is solely that of a promoter

* J. W. Mellor, "Comprehensive Treatise on Inorganic and Theoretical Chemistry," vol. 6, p. 985; London, 1940, Longmans, Green.

or catalyst. It thus becomes possible to explain the embrittlement by silicon, of platinum which is not in contact with silica but is contained in a closed system containing sulphur-bearing oil in contact with silicates at a high temperature. Volatile SiS_2 is apparently formed and serves to transport silicon from the refractory to the platinum. When the SiS_2 comes into contact with heated platinum, it is decomposed, the silicon combining with the platinum and the sulphur so liberated becoming free to combine with silicon again to form more SiS_2.

In order to obtain confirmation of the above view of the cause of embrittlement, the experiment of passing a mixture of nitrogen and carbon disulphide over platinum and platinum–rhodium wires was repeated using a tube of fused alumina in place of 'Triangle H' material. No embrittlement was found even after heating at 1200° C. for 1 hr. When wires were heated in a cavity in a graphite block with powdered sulphur at 1400° C. for 1 hr. under these conditions, however, slight attack was observed. Analysis of the graphite showed it to contain at least about 0·25 % of silicon (presumably as silicate); and since it is evident that partial combustion of the graphite would result in a concentration of silicates on the surface, it seems reasonable to consider that sufficient SiS_2 might have been formed to cause noticeable attack on the platinum.

Finally, it seemed desirable to confirm that the embrittlement produced when the wires were heated with oil in a cavity in a graphite block was of the same nature as that produced by heating in contact with carbon and sulphur. Chemical analysis of a number of fine platinum wires embrittled by heating with Mobiloil B.B. at 1400° C. for 1 hr. under the conditions of the original test, established that contamination was again caused by silicon.

Conclusions

It has long been known that if platinum or platinum alloys are heated in contact with silica or silicates in a reducing atmosphere at temperatures above about 1200° C., platinum silicides are formed at the grain boundaries which embrittle the metal. The experiments which have been described suggest that even when the platinum is not in contact with the silica-bearing material, it may be contaminated if the conditions are favourable to the formation of silicon sulphide. This compound appears to form readily in the presence of carbon and sulphur at temperatures above about 1100° C.

Thus, in order to prevent embrittlement of thermocouple wires in service, precautions should be taken to eliminate traces of lubricating oil or other materials liable to contain sulphur, from the thermocouple assembly.

Section VI—An Examination of the Microstructure of Contaminated and Embrittled Platinum and Platinum-Rhodium Wires*

By R. C. Jewell, B.Sc., A.R.S.M., A.Inst.M.M.

Synopsis

Three cases of embrittlement of platinum/platinum–rhodium thermocouples have been examined microscopically. The contamination had been caused under quite different conditions in each of the three cases. In the first case, different silicides were detected in the pure platinum and the platinum–13% rhodium wires. Severe contamination by silicon was the cause of failure, probably due to the formation of a fusible eutectic of the silicide phase and the pure platinum and possibly also of the platinum–rhodium, aided by the brittleness of the silicide phase in the case of the platinum–rhodium wire. In the second case, intercrystalline attack was clearly revealed. The constitution of the new phase could not be determined. It was, however, clearly different from either of the silicides examined in Case 1. In Case 3 the same unidentified phase was again found.

Cases are reported from time to time by the steel industry of the failure of platinum/platinum–rhodium thermocouples, chiefly due to brittleness of the platinum–rhodium wire.

It was thought that an examination of the microstructure of such contaminated and embrittled wire might throw some light on the cause of the failure.

Case 1

The very interesting case of the contamination and embrittlement of platinum/platinum–rhodium wires described in the paper of

* Reports submitted to the Liquid Steel Temperature Sub-Committee, 28th September, 1944; 12th June, 1945; and 26th January, 1946.

† Chief Metallurgist, The Sheffield Smelting Co., Ltd., Sheffield.

Mr. Manterfield of Messrs. Steel, Peech & Tozer, was the subject of this examination.

It was first observed that the platinum–13% rhodium leg of each of the three couples was embrittled near the point of failure, and in the case of two of the couples, a short length of wire broke off on bending near this point. The pure platinum leg of each couple was noticeably harder at the point of failure, and for a short distance from this point was stiffer than the remainder of the wire, which was normal and quite soft.

At the point of failure the platinum wire showed a pointed end, and in the case of one couple there was a bead or bulb which appeared to have been fused. The platinum–13% rhodium wire showed a dark crystalline fracture in each case.

One of the couples was kept for spectrographic examination, while the fractured ends of the other two couples were prepared for examination of the microstructure.

The microstructures were remarkable, and showed quite clearly that the wires had been attacked by silicon, forming a new phase of platinum silicide. The photomicrographs Figs. 16 to 18 show the severity of the contamination by silicon of the pure platinum wire. The photomicrographs Figs. 19 and 20 show the nature of the attack of the platinum–13% rhodium wire. The pure platinum and platinum–13% rhodium wires were similarly attacked, but the attack was far more pronounced in the case of the pure platinum wire. Comparison of Figs. 17 and 20 shows the difference in the severity of the attack very clearly.

In the case of the platinum–13% rhodium wire, cracks were detected in the areas contaminated with silicon. Figure 19 shows this clearly; a crack can be seen running in from the outside edge and confined to the outer contaminated area, but stopping abruptly at the edge of the uncontaminated wire. No such cracks could be detected in the pure platinum wire.

The attack of the pure platinum wire could be traced for a distance of 1 in., but in the case of the platinum–rhodium wire the attack was local and confined to the actual point of fracture.

The areas which had been attacked by silicon were distinctly porous, as can be seen in Fig. 16, indicating that a fusible eutectic had probably been formed from the platinum and the platinum silicide. This would account for the fused bead appearance of the platinum wire at the point of fracture. Figure 18 is of interest in showing the zonal contamination by silicon, the deeply etched areas being the eutectic richest in silicon; between these areas and the unattacked platinum is a narrow zone which etched more lightly, being less rich in silicon ; and finally the innermost area, which hardly etched at all, is the pure platinum unattacked by silicon.

For comparison of the microstructures, synthetic alloys were made by adding 1% of silicon to pure molten platinum and platinum–13% rhodium respectively. The microstructures of these alloys are shown in the photomicrographs Figs. 22 and 23. The silicide phase in the platinum–rhodium alloy was distinguishable under the microscope from the silicide phase in the platinum alloy, in being a harder constituent and standing out in relief with a bluish tint compared with the greyish and softer silicide phase of the platinum–silicon alloy. There was considerable segregation of the silicide phase in the platinum–rhodium–silicon alloy, but a uniform distribution of the silicide phase in the platinum–silicon alloy. Etching was effected with dilute aqua regia, the silicide phase etching readily in each case, while the pure platinum and platinum–rhodium solid solution remained unetched.

The synthetic alloys were subjected to X-ray analysis as described by Goldschmidt and Land in this symposium.

Comparison of the microstructures of these synthetic alloys containing 1% of silicon with those of the contaminated thermocouple wires under examination, confirmed that the contaminant was silicon.

Spectrographic examination of the fractured ends of one couple confirmed further that silicon was the chief contaminant, with iron, copper, and magnesium in decreasing order of importance.

Subsequently, the remainder of the three couple wires, below the point of fracture, were recovered from the solidified ingot. Examination of the microstructure of the hot junction of these couples showed that the bottom couple was in good condition, the middle couple was slightly contaminated, but the top couple was badly contaminated with silicon. Figure 21 shows the microstructure of the pure platinum leg of the hot junction of the top thermocouple, *i.e.*, that positioned near the top of the ingot. The areas attacked by silicon have etched readily, whilst the uncontaminated pure platinum remains unetched. The pure platinum wire was more severely attacked than the platinum–13% rhodium wire.

From the evidence of the microstructure, failure of the thermocouples was due to the alloying of silicon with platinum, forming a fusible phase. The silicon was derived from the fused-silica sheath, the silica probably being reduced to silicon by hydrogen or carbon monoxide given off by the

steel as it solidified. The contraction of the steel on cooling probably cracked the outer sheath, thus permitting the diffusion of gas to the inner sheath and the reduction of silica to silicon, which then alloyed with the platinum of the thermocouple wire.

Case 2

The second case of brittleness in platinum–13% rhodium wire examined was that described in the paper by Goldschmidt and Land, and referred to as contaminated wire *C*, where the thermocouple was accidentally contaminated by oil coming into contact with the wire. In this case the platinum–13% rhodium wire had fractured at two places corresponding exactly with the length of the twin-bore silica insulator. The platinum wire was unbroken.

Visual examination showed that the platinum wire had a black scaly surface near the welded junction. The wire was not brittle and did not fracture when bent by hand, but incipient cracks were detected at intervals on the surface of the wire after bending.

The platinum–13% rhodium wire had a cleaner-looking surface, but at the first fracture there was a distinct sprouty appearance, resembling a mass of fine beads, on the surface of the wire on either side of the break for a distance of about $\frac{1}{16}$ in. The colour of the wire was darker at this point, and the fracture had a dark crystalline lustre. The wire was very brittle and a piece broke off on bending, with a bright crystalline fracture.

Microsections were prepared of the platinum–13% rhodium wire and the pure platinum wire. The chief interest centres in the platinum–13% rhodium wire, which was brittle. Photomicrograph Fig. 24 shows the unetched polished section at the first fracture, 1·85 in. from the hot junction, which corresponds exactly with the length of the insulator. Intercrystalline attack is clearly seen close to the fracture and decreases further away from the point of fracture. Photomicrograph Fig. 25 shows the same area after etching, from which the intercrystalline nature of the attack is clearly evident. At points further away from the fracture the intercrystalline attack decreased and gradually receded in depth of penetration until it was confined to the outer layer only, and at a distance of about 4 mm. from the point of fracture disappeared altogether. This is shown in the photomicrograph Fig. 26, taken at a distance of 2 mm. from the point of fracture.

The fracture is directly related to this intercrystalline attack, and cracks following the grain boundary are shown in Fig. 27, where cracks starting from the edge of the wire have reached practically right across the section of the wire, which is about to break at this point.

It was not possible to identify the metallographic constituent associated with the dark grain boundary shown in the unetched polished section of the platinum–13% rhodium wire.

The microstructure of the pure platinum wire showed small intercrystalline cracks, but all were confined to the outer grains and nowhere penetrated beyond the outer surface layer. This is shown in Fig. 28. There was very little evidence of grain-boundary attack in the unetched polished section, and the intercrystalline nature of the cracks was only revealed on etching. The pure platinum wire had not been affected to the same extent as the platinum–13% rhodium wire.

From the evidence of the microstructure it is quite clear that the brittle fracture of the 13%-rhodium wire was directly associated with the intercrystalline attack due to oil having come into contact with the wire at a temperature ranging from 850° to 350° C. It is probable that the attack is due to hydrocarbon gases derived from the vaporization of the oil at this temperature, but the rapidity of the attack and consequent failure is remarkable.

Case 3

The third set of thermocouple wires were those referred to by Goldschmidt and Land as wires *E* and *F*. These wires had been used in a liquid-steel pyrometer and had failed in service.

Visual examination of the wires as received showed that the platinum–13% rhodium wire had a coarse crystalline fracture at the contaminated end, whereas the platinum wire appeared to be quite ductile and had a pointed end.

Both wires were set vertically in a mounting medium, and cross-sections were prepared for examination of the microstructure. No detectable contamination was observed in the microstructure of the pure platinum wire, but the platinum–13% rhodium wire showed a dark constituent at the grain boundaries running in from the outer surface at several points for a short distance towards the centre. Figures 29 and 30 show this quite clearly.

The constituent, which could not be identified, certainly did not etch in the same way as the platinum silicide phase described in Case 1, but appeared to be very similar to that described in Case 2, in which the platinum–13% rhodium wire was contaminated by oil.

The intercrystalline nature of the fracture in the wire is clearly revealed in Fig. 29, where

individual crystals can be seen to have broken off, leaving an incomplete section.

Conclusions

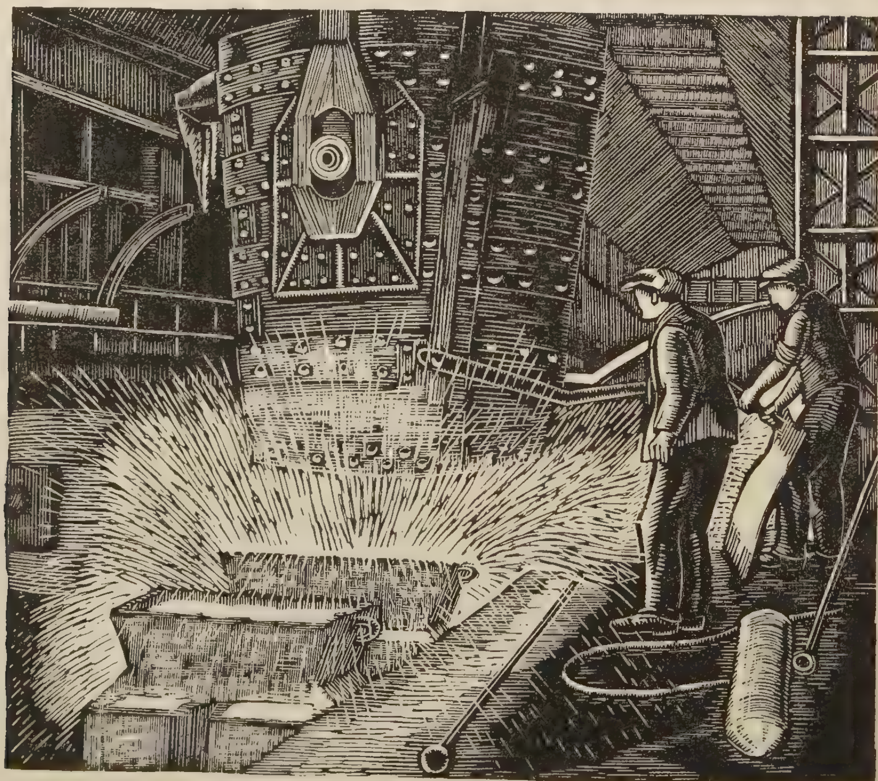
Two different silicides have been found in the thermocouple wires examined. These can be distinguished visually in the unetched polished microsection by microscopic inspection. The silicide found in the platinum–13% rhodium wire is a harder constituent, standing out in relief with a bluish tint, whereas the silicide found in the pure platinum wire is softer and presents a greyish appearance. These silicides are the same compounds detected by Goldschmidt and Land, and called by them " *R* " and " *P* " respectively. A third phase which appears to be Goldschmidt and Land's phase " *Q*," has also been detected under the microscope. Its distinctive feature is that it appears as a dark constituent and is associated with the crystal boundary. This phase is characteristic of the platinum–13% rhodium wire, but has not so far been detected microscopically in the pure platinum wire.

Two distinct types of failure were revealed. The first type was due to severe contamination by silicon, leading to the probable formation of a eutectic between the respective silicide phases and the pure platinum, and possibly also the platinum–13% rhodium. Brittleness of the silicide phase in the platinum–13% rhodium wire was a contributory factor and cracks were detected in the microstructure of this silicide phase.

The second type of failure was due to intercrystalline cracking of the platinum–13% rhodium wire, probably directly associated with the presence of a dark unidentified constituent accompanied by pronounced intercrystalline attack, as shown in one example.

Acknowledgments

The author wishes to record his indebtedness to Mr. Land, of Wm. Jessop & Sons, Ltd., and to Mr. Manterfield, of Steel, Peech & Tozer, for their kindness in sending him the samples for examination, and to the directors of The Sheffield Smelting Co., Ltd., for their permission to publish the results of this investigation.



Tundish-pouring Steel Ingots
from the original wood-cut by Viva Talbot

First Report of the Converter Refractories Sub-Committee*

Synopsis

A review of current practice is given regarding the internal shape of converters and the materials used for lining and patching. The properties of these materials have been extensively investigated in the laboratory and an effort has been made to correlate them with practical results. The detailed accounts of the drying and preheating of converter linings include the working temperature of the lining during these periods and the subsequent blowing, and the results of examination of the linings before and after use. The Report concludes with suggestions for a further programme of work on a strictly comparable basis.

Introduction

The necessity for the specific investigation of converter refractories was realised when the Side-Blown Converter Practice Sub-Committee was formed in September, 1943, to report on methods by which maximum efficiency could be obtained from existing plant.

A Panel was formed consisting of the following :

Dr. T. P. Colclough (*Convener*),
Iron and Steel Control

Dr. J. H. Chesters,
United Steel Companies, Ltd.

Dr. A. H. B. Cross,
The Brown-Firth Research Laboratories

Mr. A. T. Green,
The British Refractories Research Association

Dr. J. R. Rait,
Messrs. Wm. Jessop & Sons, Ltd.

Dr. R. J. Sarjant,
Messrs. Hadfields, Ltd.

Mr. T. H. Skelton,
Messrs. Edgar Allen & Co., Ltd.

Mr. J. D. Townsend,
Messrs. Stanton Ironworks Co., Ltd., later Messrs. Thos. Firth and John Brown, Ltd.

Mr. S. Spray (*Secretary*),
Stanton Ironworks Co., Ltd.

Dr. R. J. Sarjant was subsequently appointed Chairman.

Others who have served on the Panel are :

Mr. B. L. Collins,
Messrs. Samuel Osborn & Co., Ltd.

Mr. G. T. Hampton,
Messrs. F. H. Lloyd and Co., Ltd.

Mr. T. W. Howie,
The United Steel Companies, Ltd.

This Panel arranged and executed the work of this Report until the end of 1945, when it was taken over by the newly constituted Converter Refractories Sub-Committee, operating within the Steel Castings Division of the British Iron and Steel Research Association. The constitution of the new Sub-Committee is very largely the same as that of the old Panel, so that continuity has been maintained during the preparation of the Report. The Report now appears under the ægis of the new Sub-Committee, which expresses its thanks to those members of the old Panel who helped with the work and who are not able to assist in the publication of the Report.

Scope of the First Report

A large field of investigation has been covered in the work to date, and many apparent anomalies which have presented themselves call for much further work. It is felt, however, that a factual record of the results obtained should be made at this stage, and these are set out in the following form :

PART I—Outline of General Current Practice

PART II—Properties of Converter Lining Materials
- (*a*) *Examination of Monolithic Materials*
- (*b*) *Examination of Bricks Used for Converter Linings*

PART III—Experimental Work
- (*a*) *Drying and Preheating Studies*
- (*b*) *Study of Silica Brick Linings*

Summary

Acknowledgments

* Paper SC/CA/46 submitted by the Moulds Committee of the Steel Casting Division of the British Iron and Steel Research Association, received 20th May, 1946.

Table I (a)—*Summary of Replies to Questionnaire, Plants* A *to* E

Plant	*A*	*B*	*C*	*D*	*E*
Shape of Vessel	Oval D-shape	Circular	Oval D-shape	D-shape	D-shape
Size at Tuyeres	2 ft. 6 in. × 2 ft. 10 in.	...	3 ft. 4 in. × 3 ft. 5 in.	2 ft. 9 in. × 3 ft. 3 in.	2 ft. 6 in. × 2 ft. 9 in.
Capacity, cwt.	40	24	60	40	30
Depth of Well	6 in.	1 ft. 5 in.	1 ft. 7 in.	1 ft. 3 in.	1 ft. 5 in.
Tuyeres	6 in. × 1¼ in. splayed	5 in. × 1⅛ in.	6 in. × 1¾ in.	7 in. × 1⅝ in.	7 in. × 1¼ in.
Construction :					
Body	Monolithic	Monolithic	Silica blocks	Silica blocks	Monolithic
Nose	,,	,,	Monolithic	,,	,,
Tuyeres	,,	,,	Silica block	,,	,,
Joints	...	...	Silica cement	Silica cement	...
Expansion Joints	...	...	Silica cement felt, 4 per course	Nil	...
Drying	Air dried 2–3 days Coke fire 16–24 hr.	Air dried 7 days Coke fire 15 hr.	Gas flame 3–7 days Coke fire 24 hr.	Coke fire	Coke fire 6–7 days
Preheat	Air into remaining coke for 24 hr.	...	Coke fire with blast	...	Oil burner, to 900°–1000° C.
Charging Temperature	700° C.	1150° C.	1200° C.	900° C.	900–1400° C.
Life of Lining :					
Heats	130	80	Inner, 250–350	500	65–70
Steel produced, tons	260	96	750–1050	1000	97–105
Life of Tuyeres, heats	130	80	80–1501	70	65–70
Heats per Run	14	18	35–40	10	12
Usage of Vessel	Used alternate days	15 min. interval between heats	No interval between heats 6-hr. runs	Alternate days	...
Refractories Consumption from New Vessel, per ton of Steel, lb.	82	67	23	12¼	...
Patching	Nose made up each morning with bricks	Nose made up daily	Hot patching of belly and tuyeres mouth with ganister at end of run	Ganister between runs	Hot patching at 1350° C.
Refractories used per ton of Steel, lb.	6	33½	9¼	55	...
Nature of Wear	Immediately above tuyeres, and back to shell at nose joint on tuyere side	Severe erosion immediately over tuyeres	Severe above tuyeres for 15in. remainder fairly even. Slightly more at nose joint on belly side	Tuyere side, from tuyeres to nose	Tuyere side generally and angle of nose and body on belly side
Blowing Time, min.	12	18	9–12	22	20–25
Blowing Angle, (°)	15	15	5–10	18	New, 35–40° Old, 7–10
Metal Charge analysis, %	C 3·0, Si 1·3, Mn 1·2	C 3·1, Si 1·5, Mn 0·35	C 3·0, Si 0·1, Mn 0·4	C 2·0, Si 1·8, Mn 0·7	C ..., Si 0·8, Mn 0·5
Blown Metal analysis, %	C 0·07, Si Trace, Mn Trace	C 0·09, Si Trace, Mn Trace	C 0·08, Si 0·04, Mn 0·10	C 0·1, Si Trace, Mn Trace	C 0·08, Si Trace, Mn Trace
Additions :					
Before Blow	Nil	Nil	80 lb. of old core sand, 20 lb. of ferrosilicon (75%)	Nil	Nil
During Blow	10 lb. of ferrosilicon (80%)	Nil	Nil	Nil	Ferrosilicon (45%), nil to 150 lb. in 6 heats
Vessel Slagged	No	No	Yes	No	No
Blown Slag analysis :					
SiO_2, %	...	60·2	64·5	...	64·0
Al_2O_3, %	...	7·7	5·2	...	...
FeO, %	...	21·6	16·7	...	25·0
MnO, %	...	12·4	12·8	...	...
CaO, %	...	...	0·4	...	...
MgO, %	...	...	0·6	...	...

Plant	F	G	H	J	K	L
Shape of Vessel	D-shape	Flat on circle	D-shape	D-shape	D-shape	Circular
Size at Tuyeres	3 ft. 2 in × 3 ft. 9 in	3 ft. 4 in. dia.	2 ft. 0 in. × 2 ft. 4½ in.	2 ft. 6 in. × 3 ft. 4 in.	3 ft. 0 in. × 3 ft. 6 in.	2 ft. 6¾ in. dia.
Capacity, cwt.	70–80	40	24	40–42	60	50
Depth of Well	1 ft. 2¾ in.	1 ft. 0¼ in.	1 ft. 5 in.	1 ft. 1¼ in.	1 ft. 10 in.	2 ft. 1¼ in.
Tuyeres	8 in. × 1¼ in.	5 in. × 1½ in.	5 in. × $1\frac{5}{16}$ in., splayed	7 in. × 1¾ in.	7 in. × $1\frac{3}{8}$ in.	7 in. × $1\frac{3}{8}$ in.
Construction:						
Body	Silica blocks	Monolithic	Monolithic	Silica brick	Silica brick	Silica brick
Nose	Monolithic	,,	,,	,,	,,	,,
Tuyeres	Silica block	Silica block	,,	,,	,,	,,
Joints	Silica cement	...	...	$\frac{1}{8}$ in. × $\frac{3}{16}$ in.	Silica cement	Silica cement
Expansion Joints	4–8 in. per course	..	125 ¼-in. vents, 10–15 in. deep	$\frac{1}{16}$–$\frac{1}{8}$ in. of felt	...	Nil
Drying	Coke fire 4 days	Air dried 2 days, slow fire 3 days, blast on 12 hr., white heat 2 hr.	Air dried 12–36 hr., slow fire 3 days, blast on 12 hr.	Coke fire 48 hr.	Coke fire 14 hr. blast on 4 hr.	Air dried 7 days, coke fire 48 hr.
Preheat	Oil burner, to 900° C.	...	As above	Air into remaining coke for 1½ hr. 1100° C. (optical pyrometer)	...	Air into remaining coke for 1 hr.
Charging Temperature	...	1000° C.	1400° C.	860° C. (optical pyrometer)	800° C.	...
Life of Lining:						
Heats	Inner 90	Inner 50, full 600.	75–95	230–640	1000	425
Steel produced, tons	340	Inner 100, full 1200	90–114	460–1280	3000	1160
Life of Tuyeres, heats	90	30–40	75–95	250–300	100	75
Heats per Run	20–22	12–15	24	10–127	9	6
Usage of Vessel	8-hr. runs	15–16-hr. runs	14-hr. runs	...	30-min. heats	30-min. heats
Refractories Consumption from New Vessel, per ton of steel, lb.	...	...	115	10–12	49	4½
Patching	Hot patching with wet silica clay	Hot patching with cupola slurry	Mouth only with Sheffield blue ganister	Ganister and bricks (ganister rammed into position)	Alternate days with ganister	Every 3 days with ganister slurry and silica bricks, ganister in mouth
Refractories Used per ton of Steel, lb.	...	...	5	135	32	12¾
Nature of Wear	Tuyere side 1 ft. 3 in. above and below. 3 ft. each side of joint on belly side	1 ft. into tuyeres. 6 in. oval at nose joint	Tuyere side 9 in. belly side 6 in.	Tuyeres and body at side of tuyeres	...	...
Blowing Time, min.	15–18	20	10–12	26–28	25	30
Blowing Angle, deg.	15–20	11–12	15	29–26	14–10	18–15
	C Si Mn	C Si Mn	C Si Mn	C Si Mn	C Si Mn	C Si Mn
Charge Metal Analysis, %	... 0·6 0·5	... 1·25 0·04	3·0 1·2 0·4	3·3 1·5 0·45	2·5 1·0 0·3	3·0 1·2 0·4
Blown Metal analysis, %	0·09 Trace Trace	0·09 Trace ...	0·07 0·05 0·02	0·08 Trace 0·01	0·04 0·09 0·05	0·04 0·02 0·01
Additions:						
Before Blow	2 shovelfuls of sand, 40 lb. of ferrosilicon (75%)	Nil	Nil	Nil	...	40 lb. silica sand
During Blow	80 lb. ferrosilicon (75%)	Nil	Nil	10–30 lb. ferrosilicon (45%)	70 lb. ferrosilicon (75%)	62 lb. ferrosilicon (45%)
Vessel Slagged	Yes	No	No	No	No	No
Blown Slag Analysis:						
SiO_2, %	35·0	54·4	...	64·3	...	62·6
Al_2O_3, %	...	3·6	...	2·4	...	2·4
FeO, %	52·0	26·6	...	23·8	...	25·2
MnO, %	...	15·0	...	5·3	...	8·6
CaO, %	...	...	...	...	...	0·4
MgO, %	...	...	...	...	...	0·3

Table II (a)—*Summary of Replies to Questionnaire, Plants* M *to* Q

Plant	*M*	*N*	*O*	*P*	*Q*
Shape of Vessel	Oval D-shape	Oval D-shape	Circular	Circular	Circular
Si e at Tuyeres	1 ft. 8 in. × 2 ft. 6 in.	2 ft. 9 in. × 4 ft. 0 in.	2 ft. 2 in. dia.	...	...
Capacity, cwt.	25 to 30	40 (48–65 cwt. charged)	28 to 30.	25	2½–4 tons
Depth of Well	8 in.	1 ft. 0 in.	11 in.	...	1 ft. 1 in. to 1 ft. 6 in.
Tuyeres	4 in. × 1⅝ in.. 3 in. × 1 in., Splayed inwards	6 in. × 2 in.	5 in. × 1⅝ in.	30 in. × 6 in.	13–¼ in., 3–⅜ in.
Construction :					
Body	Monolithic	Monolithic	Silica brick	Silica brick	Silica brick
Nose	,,	,,	,,	,,	Monolithic
Tuyeres	Silica blocks	Silica blocks	,,	Fireclay (40% Al_2O_3)	Firebrick moulded
Joints	...	Ground siliceous clay	Silica cement	ganister	...
Expansion Joints	...	...	Nil	Nil	4 longitudinal ½-in. wood spacers
Drying	Coke fire 12 hr., blast on 1½ hr.	Air dried 3 days, coke fire 4 days	Coke Fire 16 hr.	Coke fire	Air dried : bottoms 5–6 days, bodies up to 3 months. Coal fire with some blast. Finally coal/coke mixture and blast
Preheat	As above to 960°–1020° C.	Pulverised coal 2 hr.	...	800° C.	
Temperature at First Charge, °C.	1100–1150	950–1000	...	500–200	1200–1300
Temperature during Day	1400–1450° C.				
Life of Lining :					
Heats	43	40	850	700–1000	1300–1450
Steel produced, tons	61	110	1250	...	4000–4500
Life of Tuyeres, heats	43	40	220	16	22–30
Heats per Run	18	11–14	12	...	23
Usage of Vessel	...	16-hr. runs	...	...	2-day runs
Refractories Consumption from New Vessel, per ton of Steel, lb.	11	249	...	4¼	35
Patching	Plastic ramming, material tamped into place	Nose	Ganister after day's run	...	Ganister between runs
Refractories used per ton of Steel, lb.	50	Nil	...	55¾	28¾
Nature of Wear	Tuyere side generally, worst from bottom to 2 ft. 4 in. above tuyeres, burning back 8 in.–12 in. at centre tuyere	Tuyere side from bottom to 18 in. above tuyeres, belly side. To joint of body and nose. All parts of nose	Fairly even generally	Tuyeres and short side worst. Build-up occurs at 45° at both sides of teeming line near top of body	Chiefly in tuyere block. Some build-up on belly side
Blowing Time, min.	14	16–22	17–20	20–25	6¾–8
Blowing Angle, deg.	63–52	12	15	Vertical	Vertical
	C Si Mn	C Si Mn	C Si Mn	C Si Mn	C Si Mn
Metal Charge Analysis, %	2·7–3·3 0·8–1·3 0·4–0·5	2·9 1·2 0·6	2·9 1·5 0·6	4·0 2·0 to 2·5 0·7 to 0·9	3·6 1·1 0·5
Blown Metal Analysis, %	0·08 0·06 0·08	0·10 0·04 0·06	0·08 0·05 0·10	0·05 Trace Trace	0·05 0·04 0·05
Additions :					
Before Blow	Nil	Nil	Nil	...	Nil
During Blow	6 lb. ferrosilicon (47%) 14–18 lb. broken glass	30–40 lb. ferrosilicon (45% Si)	20 lb. ferrosilicon (45% Si)	...	Ferrosilicon (45%) to raise temperature. Scrap to lower temperature
Vessel Slagged	No	No	No	No	No
Blown Slag Analysis :					
SiO_2, %	60–65	62	...	58–65	37–46
Al_2O_3, %	2·0–2·4	12	...	8–12	2–7
FeO, %	19·8–26·6	15	...	12–15	$\left.\right\}$ 50 to 57
MnO, %	6·7–11·1	8	...	10–15	
CaO, %	...	...	...	...	1
MgO, %	...	...	...	...	Nil

TABLE II (*b*)—*Summary of Replies to Questionnaire, Plants* R *to* W

Plant	*R*	*S*	*T*	*U*	*V*	*W*
Shape of Vessel	D-shape	D-shape	D-shape	Oval D-shape	D-shape	Oval D-shape
Size at Tuyeres	...	3 ft. 7 in. × 2 ft. 10 in.	2 ft. 5½ in. × 2 ft. 11 in.	3 ft. 2 in. × 3 ft. 8 in.	2 ft. 6 in. × 2 ft. 10 in.	2 ft. 11 in. × 3 ft. 4 in.
Capacity, cwt.	43 to 48	47	29	60 to 65	40	50
Depth of Well	1 ft. 0 in.	1 ft. 0½ in.	1 ft. 0 in.	1 ft. 3 in.	1 ft. 9 in.	1 ft. 2 in.
Tuyeres	6 in. × 1¾ in.	5 in. × 1½ in.	5 in. × 1½ in.	7–2 in.	5–1⅞ in.	5
Construction: Body	Monolithic	Bricks, black silica	Bricks, black silica	Outer, monolithic; Inner, silica brick	Monolithic	Silica blocks
Nose	,,	,,	,,	Monolithic	,,	Silica blocks
Tuyeres	,,	Silica	Silica	Silica blocks	,,	Silica bocks
Joints	...	...	...	Silica cement	...	Milled ganister
Expansion Joints	...	⅛ in. of felt every 18 in.	...	Corrugated sheeting next to shell	½-in. vent rods 6 in. apart	...
Drying	Air dried 7 days (min.), slow fire 2 days	Coke fire 30–32 hr., blast on 60–90 min.	12-in. town's gas flame till lining shows dull red, then coke fire and blast	Air dried several days, coke fire at least 7 days, intermittent blast last day	Small gas flame, 24 hr., coke fire 8 hr., blast 2 hr.	Air dried, 8 hr., coke fire 2 ft. above tuyeres for 6 hr. body filled with coke and blast on for 2 hr. More coke added and blast on 2 hr.
Preheat	Coke fire	As above	...	Coke fire, intermittent blast for 2–3 hr.		
Temperature at First Charge, °C.	1190	1300	1200–1300	1450	1260	1050–1340
Temperature during Day	...	1430–1500	...	...	...	...
Life of Lining: Heats	160–260	23	80	(Inner) 150	700–800	340
Steel produced, tons	300–580	54	120	470	...	850
Life of Tuyeres, heats	160–260	23	55	70–80	20	80
Heats per Run	...	12–14	12	15	...	20
Usage of Vessel	...	16-hr. runs	6–8-hr. runs	16-hr. runs	...	3–5-day runs
Refractories Consumption from New Vessel, per ton of Steel, lb.			85	45½	11	15
Patching	...	Mouth only	Wet slurry on teeming side every 12 heats. Hot patching on tuyere side	Nose made up with ramming material, slurry on bottom	Hot- and cold-patched with siliceous clay	Back: hot-patched Belly; cold-patched
Refractories used per ton of Steel, lb.	...	...	49	34	50	16¾
Nature of Wear	...	Tuyere side generally. Pouring side, above tuyeres	Tuyere side and trunnion sides	2 ft. above and 9 in. below tuyeres. 3 ft. either side of joint on belly side	10 in. tuyere side 8 in. trunnion side 4 in. pouring side	Tuyere side. 1 ft. 2 in. above and below tuyeres Belly, 1 ft. above and below centre line of tuyeres Nose general
Blowing Time, min.	16	19–22	8–11	13–14	15–16	33
Blowing Angle, deg.	18	10–12	10–12	15–17	15	15
Charge Metal Analysis, % (C / Si / Mn)	0·05 / ... / ...	3·0–3·4 / 0·9–1·1 / 0·2–0·4	2·8–3·2 / 0·4–0·5 / 0·5–0·7	2·5 / 0·3–0·4 / 0·5	3·0 / 1·5 / 0·5	3·2 / 1·1 / 0·74
Blown Metal Analysis, % (C / Si / Mn)	... / ... / ...	0·04–0·08 / 0·03–0·08 / 0·03–0·07	0·07–0·13 / 0·06–0·08 / 0·08–0·09	0·1 / 0·1 / 0·05	0·07–0·10 / 0·07 / 0·07	0·06 / 0·05 / 0·11
Additions: Before Blow	Nil	Nil	Nil	6–8 shovels of sand	Nil	Nil
During Blow	Nil to 60 lb. Ferrosilicon (75%)	Nil to 25 lb. ferrosilicon (75%)	20–30 lb. ferrosilicon (75%)	Nil	Up to 20 lb. ferrosilicon (45%)	Nil
Vessel Slagged	Yes	Yes	Yes	Yes	No	Yes
Blown Slag Analysis: SiO_2, %	...	55–60	60–63	...	66	61·3
Al_2O_3, %	...	0·3–1·5	4–5	...	...	12·7
FeO, %	...	18–25 (Fe_2O_3 5–20)	20–29	...	33·5	19·1
MnO, %	...	2·5–7	9–11	...	...	5·8
CaO, %	...	0·07–0·23	0·5–0·6	...	...	0·7
MgO, %	...	...	...	...	...	...

Part I—OUTLINE OF GENERAL CURRENT PRACTICE

In order to obtain information on current practice as a starting point in the investigation on converter linings, a questionnaire was circulated to steel foundries, and replies were received from 22 firms. The Panel very much appreciate this co-operation and due acknowledgment to these firms is made in a later stage of the Report.

The essentials of the details are presented in collected form in Tables I and II. It is difficult to generalize on the construction, operation, and life of linings, and Table III shows that variations in all aspects are considerable (*see* column 2). For purposes of comparison the plants have been separated into three main groups according to capacity, *i.e.*, 23–30 cwt., 40–47 cwt., and 50–60 cwt. (*see* columns 3, 4, and 5). Outside this grouping is one plant of 70–80 cwt. capacity, and two bottom-blown converters, details of which it was originally thought might possibly be of use in certain respects.

This analysis shows a preference for monolithic linings in small vessels, gradually changing to brick linings for the largest vessels. As is to be expected, the drying time increases with size, and a point of interest is that the number of heats blown during a run is almost identical, irrespective of capacity. In other words, it appears that the factor for deciding capacity is the ability of the foundry to take approximately 15 heats of steel daily.

It is difficult to discuss converter operation without departing from the strictly refractories viewpoint, but attention may be called to the general average blowing angle of 15°. The big departures from this are mostly due to plant conditions. It is definitely indicated that the larger vessels are the more economical from the standpoint of refractories consumption, despite the fact that total blowing time is in the reverse order.

Lining Wear and Patching

Line-drawings of converter plants are shown in Fig. 1. The shaded portion indicates the extent of wear. It will be noted that the wear is extensive on the blowing side, particularly in the region of the tuyeres, and is more pronounced on the upper than on the lower side. Wear also takes place on the teeming side, mainly from the junction of body with nose and thence to the nose end. Ingenuity has been shown in some cases in designing the lining with a thicker section at the points of maximum wear, in addition to the generally

Table III—*Details of Converter Design and Operation Affecting the Life of Linings*

Vessel Capacity	Variations	General Trend with Vessel Capacity		
	24–80 cwt.	23–30 cwt.	40–47 cwt.	50–60 cwt.
		6 plants	8 plants	5 plants
Vessel Shape	Circular, D-shape, oval	D-shape	D-shape	D-shape
Depth of Well	6 in.–2 ft. $1\frac{1}{2}$ in.	1 ft. 2 in.	1 ft. 2 in.	1 ft. 7 in.
Number of Tuyeres	5 to 8	5	6	6
Size of Tuyeres, in.	$1\frac{1}{8}$ to 2	$1\frac{1}{4}$	$1\frac{5}{8}$	$1\frac{5}{8}$
Tuyere Arrangement	Right-angled—splayed	Right-angle	Right-angle	Right-angle
Lining Material	Brick, monolithic, both	Monolithic	Monolithic and brick	Brick
Drying and Preheating Practice	$13\frac{1}{2}$ hr.–9 days	$4\frac{1}{2}$ days	$4\frac{1}{2}$ days	6 days
Vessel Temperature	700° C.–1400° C.	1260° C.	1100° C.	1150° C.
Number of Heats per Run	6 to 24	16	12	16
Blowing Time, min.	8 to 33	16	19	22
Blowing Angle, (deg.)	5 to 63	15	16	15
Life of Lining with Patching, Tons of Steel Produced :	54 to 3,000	95	600	800
Refractories Consumption :				
Lining, lb. per ton of steel	$4\frac{1}{4}$ to 115	70	30	27
Patching, lb. per ton of steel	5 to 135	35	50	21
Total, lb. per ton of steel	17 to 146	105	80	48

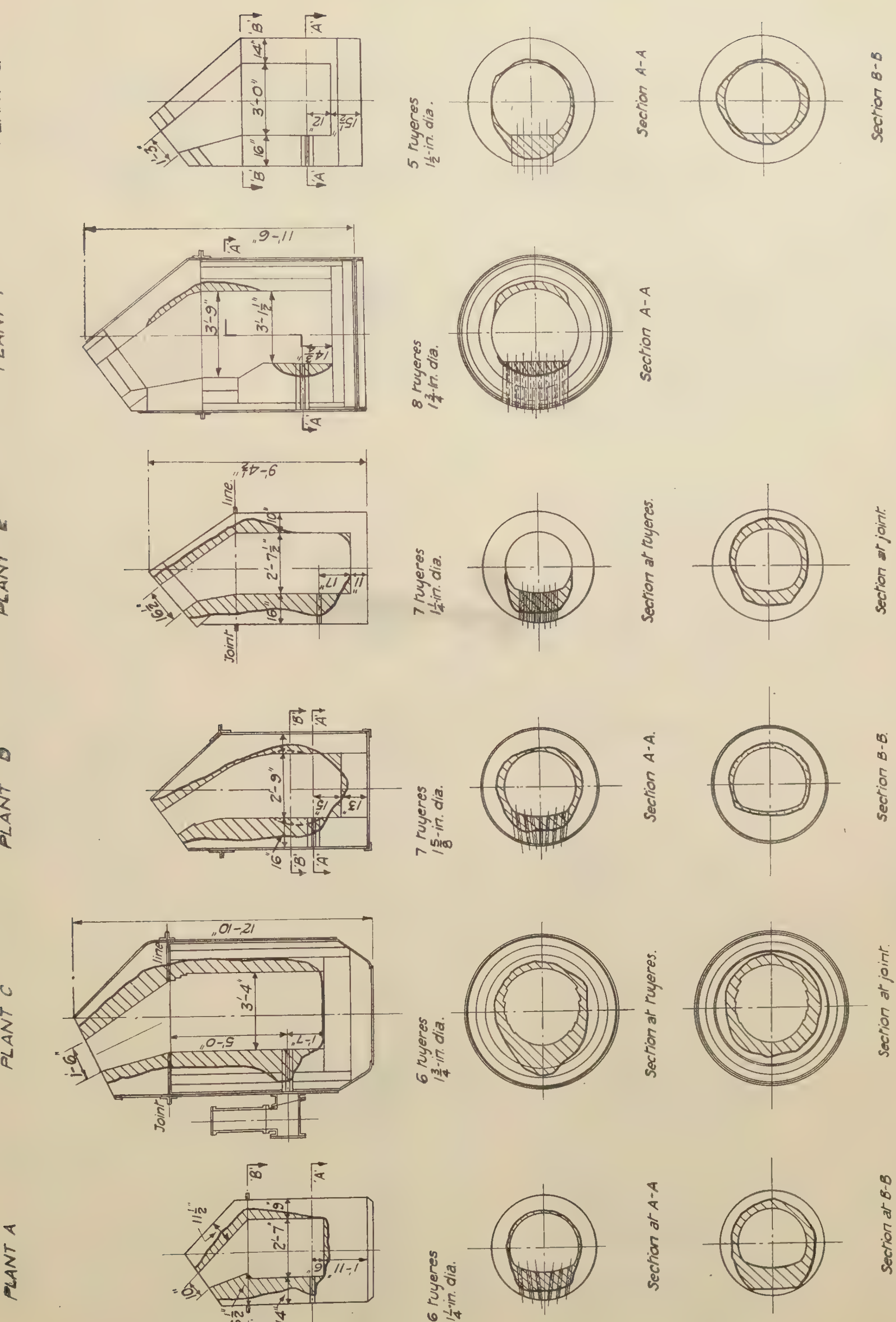


FIG. 1 (a)—Details of converter construction at Plants A to G

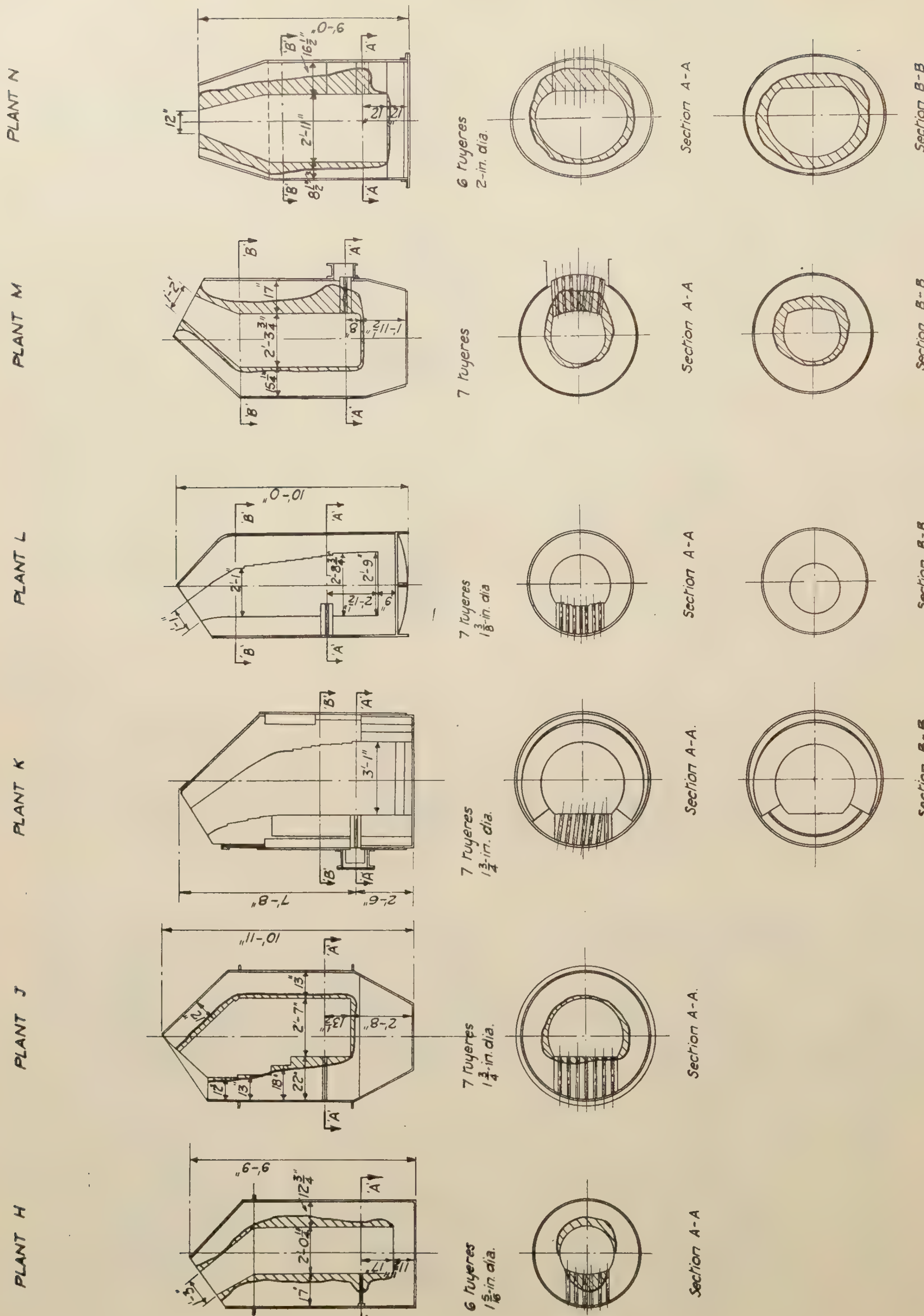


FIG. 1 (*b*)—Details of converter construction at Plants *H* to *N*

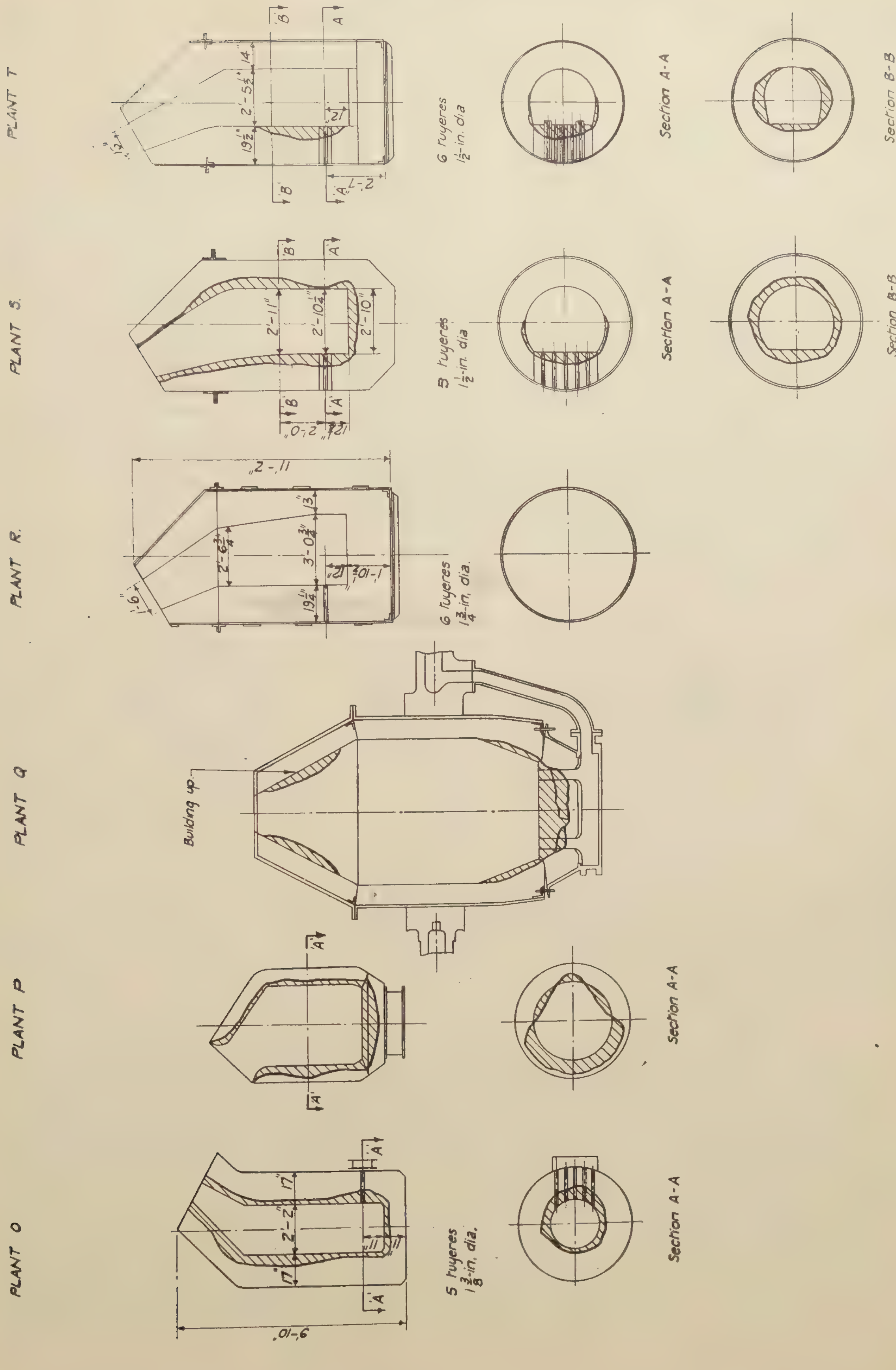


FIG. 1 (c)—Details of converter construction at Plants O to T

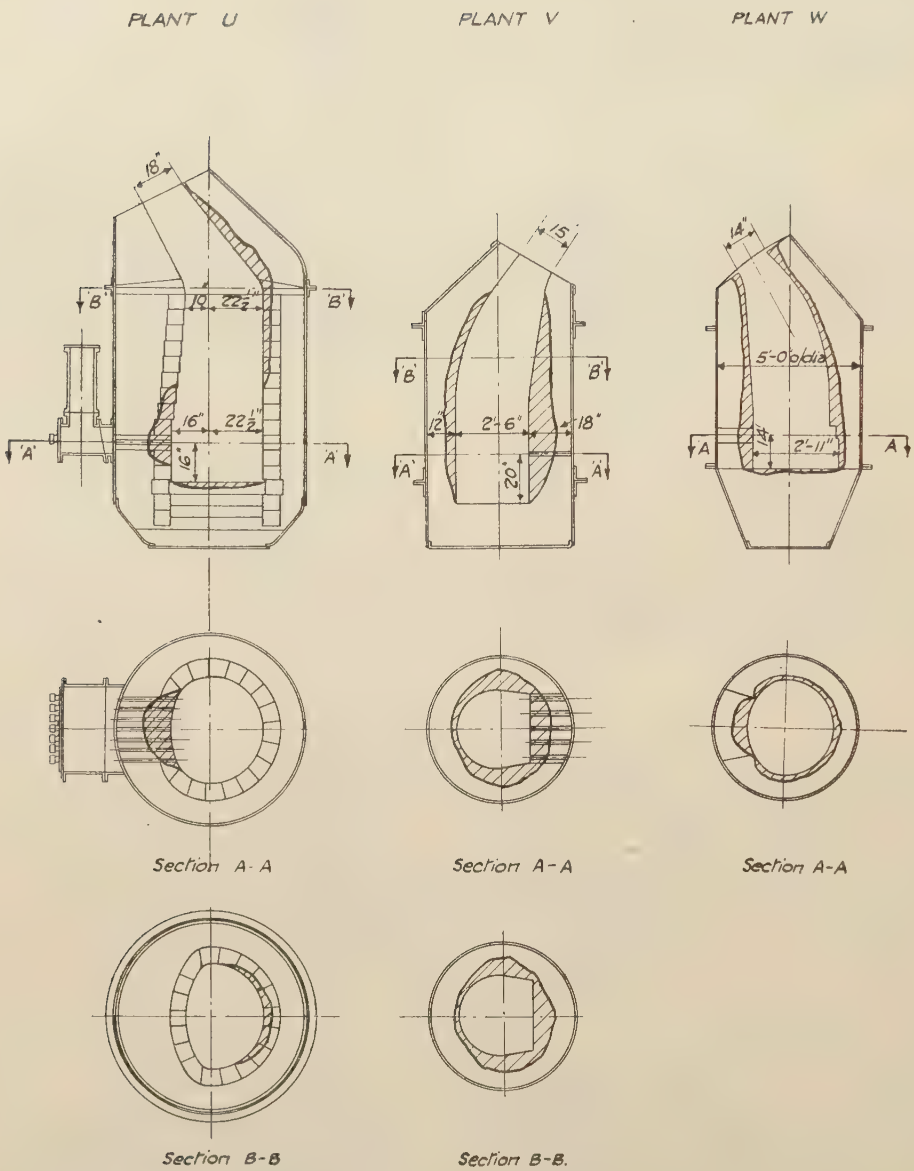


FIG. 1 (d)—Details of converter construction at Plants U, V and W

adopted practice of a thicker lining on the tuyere side.

The first study of lining life in terms of number of heats or tons of metal converted, suggested that this was in some cases being weighted by the method and amount of repair, and replies to a further questionnaire on patching practice showed that this varied considerably. The extremes were, (*a*) no patching at all, the converter being taken out of service when the inner 'veneer' lining was worn through, and (*b*) bringing back the lining to its original shape and size after each run. The intermediate stages involved hot patching in varying amounts.

A substantial improvement in lining life was effected at one plant by charging sand into the converter with the iron, to provide an acid oxide to combine with the iron oxide produced in the early stages of the blow. It was necessary from a theoretical standpoint, as this plant was using very low silicon iron. A few other plants using iron of normal silicon content later adopted the practice. Their method of operation, particularly with longer intervals between heats, may have resulted in some fritting of the sand to the lining. The sand addition may be considered as a refractories consumption, in so far as its use resulted in longer lining life, but the very low cost must be borne in mind, particularly with the use of old core sand which would otherwise be thrown away.

The technique of hot patching referred to above, varies considerably from plant to plant. In some cases the material is mixed with water to a creamy consistency and applied to the part to be repaired immediately after the last metal is tapped from the converter. At other works the vessel is allowed to cool to 600° C., and the patching material, with a water content of 15%, is tamped into place.

In the other method of patching, *i.e.*, the building up to approximately original size, the repairs are carried out when the vessel has cooled off sufficiently to allow a man to work comfortably inside. Ganister, etc., of normal consistency is used, and in some cases pieces of broken brick are bedded in.

It is not possible to draw conclusions from the data available as regards the result of each method in terms of efficiency. Each has its own theoretical and practical advantages, and plant conditions are obviously the deciding factor. Some work of a strictly comparable nature is called for, as it should be possible to even out the large variables reported, *i.e.*, 5–135 lb. per ton of steel.

The materials used for lining side-blown converters can be conveniently classified as (*a*) monolithic linings and (*b*) brick linings. It is not considered possible at present to assess the relative merits of monolithic and brick linings in view of the large variations in converter practice which exist, but the Panel thought it desirable to put on record the results of its examination of various monolithic materials and bricks, as a basis for comparison in future work.

The monolithic materials consist essentially of quartz in the form of ganister, quartzite, or sand, with varying proportions of clay added as a bonding medium. The bricks are of the type normally supplied for open-hearth-furnace roofs, although one quality with a high iron oxide content is specially manufactured for converters. Five samples of monoliths supplied, and a number of bricks used in converter linings were examined.

Part II—PROPERTIES OF CONVERTER LINING MATERIALS

Sect. A—Examination of Monolithic Lining Materials

Chemical Analysis

Analyses of the five materials tested were submitted to the Panel by two members. The results showed certain discrepancies due to the difficulty of obtaining representative samples for analysis. The analyses given in Table IV are put forward as average compositions of the materials in question.

Grading and Refractoriness

The grading was determined by mechanical sieving after removal of the clay fraction by the A.F.A. decantation method. The refractoriness of the clay grade was determined under reducing conditions in a carbon-resistance tube furnace, whilst the refractoriness of the material in bulk

Table IV—*Chemical Analyses of Monolithic Materials*

Constituent	Material				
	V	*M*	*A*	*G*	*E*
SiO_2, % ...	88·0	91·5	89·0	90·5	94·0
Al_2O_3, % ...	6·0	3·5	6·3	4·5	2·0
Fe_2O_3, % ...	1·5	1·1	0·9	1·3	0·5
TiO_2, % ...	0·4	0·7	0·5	0·4	0·2
CaO, % ...	0·9	0·4	0·4	0·4	1·1
MgO, % ...	0·2	0·3	0·2	0·3	0·2
Loss on ignition	2·6	1·6	2·3	1·8	1·1
Total ...	99·6	99·1	99·6	99·2	99·1

Table V—*Grading and Refractoriness of Monolithic Materials*

		Material				
		V	*M*	*A*	*G*	*E*
Moisture Content as received, %		9·0	7·5	6·0	4·0	7·0
Grading (B.S.I. sieves):	On 1 in.	...	...	...	...	8·1
	1 in.–½ in.	...	...	...	...	23·1
	½ in.–¼ in.	...	...	...	5·7	17·3
	¼ in.–7 mesh	1·7	7·6	3·0	22·7	14·4
	7–25 mesh	0·9	7·7	3·6	22·4	11·9
	25–72 mesh	1·2	4·3	1·8	8·6	2·8
	72–100 mesh	6·8	1·2	17·4	5·0	2·2
	100–150 mesh	31·4	3·1	19·2	4·5	2·1
	150–300 mesh	36·3	19·7	14·8	9·0	8·9
	Through 300 mesh	3·7	21·6	19·1	10·2	0·7
	Clay grade	18·0	34·8	21·1	11·9	8·5
		100·0	100·0	100·0	100·0	100·0
Refractoriness, °C.:						
Bulk, °C.		1680	1670	1690	1680	...
Clay grade (reducing atmosphere—from start to soften to end point), °C.		1600 to 1700	1500 to 1650	1580 to 1715	1460 to 1515	...

Table VI (a)—*X-Ray Examination of Monolithic Lining Materials* V *and* M

Grading (B.S.I. Sieves)	Percentage on Sieves: Total	Percentage on Sieves: Type	Type	Constitution
			Material V	
¼ in.–7 mesh	5·1	...	Bastard ganister	Quartz + trace of unknown
7–25 mesh	2·2	...	,, ,,	Quartz + very small amount of unknown, possibly felzspar
25–72 mesh	1·5	...	...	Quartz
72–150 mesh	40·1	...	Sand grain	Quartz
Through 150 mesh	51·1	...	Average sample	Quartz + small amount of kaolinite
		5–10 (approx.)	Portion having low settling rate	Kaolinite + small amount of quartz
			Material M	
¼ in.–7 mesh	11·7	10	Pebbes	Quartz
		<1	Sandstone-like	Quartz
		<1*	Black shiny	Graphite
		<0·5	White lumps, soft	Quartz
		<0·5	Grey lumps, soft	Kaolinite + small amount of quartz
7–25 mesh	7·8	7	Pebbles, broken	Quartz
		<1	Irregular shaped lumps, brown	Quartz + hydrated iron oxide
		<0·5	White lumps, soft	Hydrated calcium sulphate
25–72 mesh	7·8	...	Average sample	Quartz
72–150 mesh	6·5	...	Sand grains	...
Through 150 mesh	66·2	...	...	Quartz
		5 (approx.)	Portion having low settling rate	Quartz + small amount of felspar and mica

* The presence of this lump of flaked graphite is possibly accidental

TABLE VI (b)—*X-Ray Examination of Monolithic Lining Materials* A, G, *and* E

Grading (B.S.I. Sieves)	Percentage on Sieves: Total	Percentage on Sieves: Type	Type	Constitution
		Material A		
¼ in.–7 mesh ...	7·1	6	Pebbles, broken	Quartz
		<0·5	Sandstone-like	Quartz
		<0·5	Hard lumps, brown	Quartz
		<0·5	Hard lumps, black	Quartz
7–25 mesh	6·0	<5–6	Pebbles, broken	Quartz
		<0·5	Hard lumps, brown	Quartz
		<0·5	Soft lumps, white	Calcite + quartz
25–72 mesh	3·6	...	Average sample	Quartz
72–150 mesh ...	44·0	...	Sand grains	...
Through 150 mesh ...	39·3	...	Average sample	Quartz + small amount of clay
		...	Portion having low settling rate	Kaolinite + quartz
		Material G		
¼ in.–7 mesh ...	29·5	14–15	Grit-like, hard, grey	Quartz
		14	Grit-like or quartz-like, hard, white	Quartz
		<0·5	Grey lumps, soft	Kaolinite + small amount of quartz
		<0·5	Lustrous quartzite, hard ...	Quartz
7–25 mesh	20·5	20	Silica rock as above	Quartz as above
		0·5	Grey lumps, soft	Kaolinite + small amount of quartz
25–72 mesh	9·0	...	...	...
72–150 mesh ...	12·4	...	Average sample	Quartz
Through 150 mesh ...	28·6	...	...	...
		1–2	Portion having low settling rate	Quartz + mica
		Material E		
On ½ in.	31·6	...	Quartzite-like, hard	Quartz
½ – ¼ in.	15·4	...	As above...	Quartz
¼ in.–7 mesh ...	15·7	...	As above...	Quartz
7–25 mesh	11·8	...	...	...
		0·5	Irregular lumps, dark	Calcite + quartz
25–72 mesh	2·0	...	...	...
72–150 mesh ...	8·8	...	...	Quartz
Through 150 mesh ...	14·7	...	...	...
		<1	Portion having low settling rate	Quartz + small amount of calcite

was determined by the usual method used for refractory materials. The results of these tests are given in Table V.

X-Ray Examination

Further samples of the monolithic lining materials were graded and the fractions examined by the X-ray powder method for constitution, and by the pinhole method for crystalline texture or type. The results are given in Table VI.

Behaviour on Firing

Standard A.F.A. specimens (2 in. dia. × 2 in. high) were prepared, and rammed with 50 blows in the A.F.A. ramming machine. In the case of material No. 6 an A.F.A. specimen could not be prepared owing to its coarse texture. For this material a block 4 in. sq. × 2½ in. thick was used. Bulk-density measurements were made in the as-rammed, the dried, and the fired states. Two

firing treatments were given, (*a*) 1410° C. for 2 hr., and (*b*) 1530° C. for 2 hr. The rate of heating to the required temperature was controlled at 5° C./min. Two sets of specimens were examined from separate firing trials at 1410° C. The results of these tests are given in Table VII. The shrinkage and expansion figures given are the average of the linear changes in diameter and length.

Discussion of Results

Material V—This has the finest grading of the samples examined. It consists of quartz derived from a relatively soft silica rock and appears to contain material actually added as sand grain. The clay bond has a relatively high refractoriness.

Material M—This material is of relatively high purity and contains a high proportion of very fine silica. It is the most volume-stable at 1530° C. of the materials examined.

Material A—The grading of this material is relatively fine and it appears to contain a fair amount of reasonably refractory clay, which causes a shrinkage on firing at 1530° C.

Material G—This material has been prepared from two distinct types of silica rock and is graded to give a high packing density. The impurity content is very small and the material still shows an expansion on firing at 1530° C.

Material E—This material is extremely coarse and appears to consist almost entirely of crushed quartzite rock. It shows little sign of fusion on

Table VII—*Behaviour of Monolithic Lining Materials on Firing*

	Material				
	V	*M*	*A*	*G*	*E*
Moisture Content, % ...	8·7	7·5	5·8	5·9	9·0 (4 in. × 4 in. × 2½ in. specimen)
Drying Shrinkage, % (average of 6 specimens)	0·34	0·24	0·33	0·62	0·13
Firing Expansion, % (2 hr. at 1410° C., average of 2 tests)	1·2	2·6	1·9	3·1	2·3
Firing Expansion or Shrinkage, % (2 hr. at 1530° C.)	−3·9	+0·3	−1·2	+4·4	n.d.
Appearance after Firing at 1410° C.	Very little cracking	Showing iron spots. Badly cracked on one side	Cracked on top edge of specimen	Incipient fusion between the grains of the coarse base material	No signs of fluxing or cracking
Appearance after Firing at 1530° C.	Cracked, and showing incipient surface fusion	Cracked badly	Numerous fine cracks but little deformation	Showing cracks and incipient fusion. No deformation	n.d.
Bulk Density :					
Wet, g./ml.	1·95	2·04	1·97	2·13	Approx. 2·30
Dried (at 130° C.), g./ml. ...	1·78	1·89	1·85	2·01	2·08
Fired (at 1410° C.) :					
1st set, g./ml.	1·83	1·72	1·71	1·82	1·87
2nd set, g./ml.	1·70	1·79	1·74	1·91	...
Apparent Porosity when fired at 1410° C., % :					
1st set	26·9	29·0	32·7	26·8	...
2nd set	33·5	28·3	33·3	25·1	...

n.d. = Not determined

firing at 1410° C. due to its coarse nature and relatively high degree of purity.

General

There is considerable variation in the grading of these materials. The very coarse nature of Material *E* is noteworthy and it appears to contain comparatively little clay. The clay grades are generally related to the alumina contents as found by chemical analysis, although it should be pointed out that in the case of Material *A* this grade contains a high proportion of very fine silica, and therefore it may be misleading to estimate the clay content from a determination of the clay grade.

The refractoriness of the clay grades in Material *G* is low. It appears that the proportion and nature of the clay added is just sufficient to knit the grains together without inducing deformation.

All the materials examined show an expansion on firing at 1410° C. due to the conversion of the raw quartz present. On firing at 1530° C., a smaller expansion is obtained except in the case of Material *G*. Whether an expansion or a shrinkage will be obtained will depend on the amount and nature of the clay present, rate of conversion of the quartz, grain-size, etc., and hence it is not surprising that there is no obvious relationship between the chemical analysis and the linear change on firing, although the clay content does seem to be an influencing factor.

Conclusions

As materials of so widely different properties as those examined are in fact all used with at least some degree of success for converter linings, it would appear that operational factors are of paramount importance in controlling refractories consumption. Certain general conclusions can, however, be drawn from the examination of these materials :

(1) Over 90% of silica appears to be desirable, and the clay added should be sufficiently fine to develop adequate plasticity when present in relatively small amount.

(2) Medium and coarser gradings give the highest packing densities, and other things being equal, should behave better in service than material of lower packing density.

(3) The relative merits of different types of

Table VIII—*Properties of Silica Bricks used in Side-Blown Converters*

Brick Manufacturer	1		2	3	4(*a*) (Specially high iron)		5
Type	Converter	Ordinary	Ordinary	Ordinary	Slop-Moulded	Rammed	Converter
Chemical Analysis :							
SiO_2, % ...	96·25	96·17	94·67	96·44	92·10	91·81	96·16
Al_2O_3, % ...	1·43	0·31	1·20	0·53	0·92	0·83	0·84
Fe_2O_3, % ...	1·08	1·22	1·30	0·75	3·46	2·99	0·75
FeO, % ...	...	...	...	...	1·00	1·86	...
TiO_2, % ...	0·22	0·52	0·27	0·47	0·10	0·10	0·20
CaO, % ...	0·99	1·76	1·83	1·96	1·59	1·62	2·29
MgO, % ...	0·14	0·08	0·11	0·07	0·12	0·10	0·06
Loss on Ignition, % ...	0·10	0·05	0·06	0·11	0·09	0·07	0·02
Bulk Density, g./ml. ...	1·69	1·80	1·84	1·68	1·78	1·88	1·71
Specific Gravity ...	2·33	2·36	2·36	2·37	2·45	2·36	2·34
Apparent Porosity, % ...	27·40	22·60	21·90	28·20	25·80	18·30	27·00
After Expansion, % ...	0·30	0·86	...	1·02	−0·01	Nil	...
2 hr. at temperature, °C. ...	1410	1410	...	1500	1500	1500	...
Refractoriness, °C. ...	1690	1710	1670	1710	1700 to 1710	1700 to 1710	1710
Thermal Expansion :							
Room temperature to 100° C., % ...	0·136	0·145	...	0·162	0·128	0·142	0·159
" " 200° C., % ...	0·587	0·588	...	0·650	0·546	0·565	0·584
" " 300° C., % ...	0·907	0·970	...	1·035	0·791	0·796	0·929
" " 500° C., % ...	1·097	1·143	...	1·205	0·988	0·989	1·101
" " 600° C., % ...	1·153	1·249	...	1·270	1·043	1·044	1·184
" " 1000° C., % ...	1·237	1·330	...	1·367	1·132	1·130	1·250
" " 1200° C., % ...	...	...	...	...	1·084	1·098	...

Table IX—*Properties of Silica Bricks made by Suppliers of Acid-Converter Linings*

Brick Manufacturer	1	2	3	4 (*b*)	5	6
Apparent Porosity, %	28·90	22·80	22·70	25·40	...	...
Bulk Density, g./ml.	1·66	1·83	1·79	1·73	...	...
Specific Gravity	2·34	2·37	2·35	2·31	2·34	2·52
Cold Crushing Strength, lb./sq. in. ...	4000	...	...	...	...	...
Permeability, c.g.s. (perpendicular to 9 in. × 3 in. face, through 1 skin)	0·142	0·041	0·049	0·079	...	...
After Expansion, % (2 hr. at 1500° C.) ...	1·8	0·48	1·1	0·2	...	6·7
Refractoriness–under-load, 25 lb./sq. in. (Maintained at 1600° C.)	Failed after 56 mins.	Failed after 24 mins.	0·7% Exp. after 1 hour	0·6% collapse after 1 hour	...	...
Spalling Resistance Index (450° C. test)	$\left.\begin{matrix}1\\1\end{matrix}\right\}1$	$\left.\begin{matrix}16\\19\end{matrix}\right\}18$	$\left.\begin{matrix}2\\2\end{matrix}\right\}2$	$\left.\begin{matrix}2\\1\end{matrix}\right\}1$	$\left.\begin{matrix}1\\1\end{matrix}\right\}1$	50+
Refractoriness, °C.	1690	1710	1710	1710	1710–1730	...
Remarks on Microscopical Examination	...	Appreciable amount of raw quartz. Good tridymite development	Appreciable amount of raw quartz, which is very variable in crystal size. Tridymite development rather poor	No raw quartz. Tridymite moderately well developed	...	Contains about 75% of raw quartz
Normal use	Open-hearth furnace roofs					Acid-bessemer vessel lining

monolithic lining materials can only be proved by extended trials in the same plant.

Sect. B—Examination of Bricks Used for Converter Linings

In Table VIII the results of laboratory tests on silica bricks supplied for converter linings are given. Table IX gives the results of tests on silica bricks supplied for open-hearth-furnace roofs. It is apparent from an examination of these Tables that in general the quality of the silica bricks used for converter linings is the same as that used for open-hearth-furnace roofs. The only exception is the special high iron quality 4 (*a*), which is apparently manufactured specifically for use in converters. The high iron content promotes tridymite formation and so increases the spalling resistance by decreasing the thermal expansion occurring below red heat.

As in the case of the monolithic lining materials examined, there is a wide range of properties in these bricks, although all are used successfully in converter linings, again emphasizing the importance of operational factors in influencing consumption of refractories.

Apart from the specially prepared brick 4 (*a*), the property which shows the widest variation is the specific gravity. This is controlled by the firing treatment in the kiln and has a major influence on the thermal expansion and thus on the thermal shock resistance. Bricks which are soft-fired (high specific gravity) have a much higher thermal shock resistance than bricks which are hard-fired. It is likely therefore that the conditions during the heating-up of a new converter lining will largely determine the degree to which hard-fired bricks can be successfully used. Soft-fired bricks will continue to expand at temperature due to further conversion, although this growth will be modified by the fluxing action of FeO and MnO in service.

As with monolithic linings, the relative merits of different types of silica bricks can only be assessed by extensive trials in one plant under controlled conditions.

Part III—EXPERIMENTAL WORK

The replies to the questionnaires indicated that varied conditions existed in different foundries throughout the whole thermal history of converter linings. This suggested that more detailed knowledge of these conditions and their effect on the stability of the lining would provide useful information regarding the following :

(i) The rate and method of drying.

(ii) After drying, the maximum rate of preheating, taking adequate account of the properties of the lining materials and the subsequent speed of raising to working temperature.

(iii) The temperature distribution through the lining during drying, preheating, and blowing.

(iv) The temperature distribution change with sequential blowing of charges.

(v) The changes of chemical and physical characteristics in the lining during service.

Such information is necessary both for brick and monolithic linings. The work described below relates only to brick linings ; similar data have yet to be obtained from monolithic linings.

The experimental work was divided into two sections :

(*a*) Drying and preheating studies of brick linings. At two plants, the temperature distribution during blowing was also studied.

(*b*) Study of silica-brick linings after service.

Sect. A—Drying and Preheating Studies

Records of the temperature distribution changes within silica-brick converter linings have been obtained from three different plants, *K*, *C*, and *T*.

Plant *K*

A preliminary study during the preheating of vessels after patching showed that a rapid rise to about 160° C. occurred 1 in. behind the inner face of the lining. The patched vessel then showed a gradual rise of temperature up to 620° C. during 11 hr. with a gentle blast applied to a burning coke bed. After retuyering the initial rise of temperature was also rapid. Following the lag at 160° C. the temperature rose from 200° to 440° C. in an hour and then climbed very gradually to 590° C. in a further 14 hr.

No spalling of the brickwork was reported in either case, but it is evident from these times and temperatures that the preheating period was not being employed to the best advantage and that the final temperature attained was too low for immediate use of the vessel. Under these circumstances only 200° C. was reached $5\frac{1}{2}$ in. behind the hot face at the end of the preheating period.

Plant *C*

The drying and initial preheating of vessels was carried out by means of gas flares at this plant ; the intermediate preheating by means of coke fires with natural draught ; and the final preheating before blowing, by applying an air blast to the coke.

Thermocouples were placed 1 in., 6 in., 11 in., and 21 in. (at shell) from the inner face of the lining, and records of the temperatures were obtained using a potentiometer-type recorder. Figure 2 shows the records obtained, re-drawn to more convenient scales from the original.

Coke was charged $44\frac{1}{2}$ hr. after drying by the gas flare had commenced, when the temperature distribution was :

Couple Position	Temperature, °C.
1 in. from hot face.	640
6 ,, ,, ,, ,,	370
11 ,, ,, ,, ,,	190

A rapid fall of hot-face temperature followed the introduction of the coke, necessitating the application of the gas again to ensure ignition of the coke. This caused a delay of 3 hr. and the hot-face temperature then rapidly rose 200°/hr. to 900° C., and became fairly stable around 970° C. Application of the air blast brought the lining temperature up to 1320° C. in 35 min., when the blast was shut off and re-introduced for 10 min. shortly before the first metal charge. The hot-face temperature fell from 1300° to 1210° C. between the emptying of the vessel of coke and charging the metal.

The later portion of the record shown in Fig. 2, indicates the fluctuation of hot-face temperature during the blowing of three heats. Then an unfortunate accident destroyed the couple installation.

Plant *T*

At this plant, records were obtained covering the initial drying and preheating stages and the subsequent blowing of 70 heats with interspersed preheatings. Eighteen heats were blown before failure of the hot-face couple occurred, and the records were continued using the remaining couples. Owing to plant requirements at the time these tests were made, it was not possible to put the vessel into service as soon as it could be

preheated to a suitable temperature, and the preheating was therefore unduly prolonged.

A gas flare was used for drying, but this was too small to be of use for preheating the lining, which was done by means of coke, using natural draught except just before blowing.

Four couples were installed on the left-hand trunnion side (facing blast box), $13\frac{1}{2}$ in. above the centre-line of the tuyeres and at $\frac{1}{2}$ in., 5 in., 10 in., and 17 in. distance from the hot face of the lining, as shown in Fig. 3. A 6-line potentiometer-type recorder, scaled 0–1750° C., was used throughout. The whole lining of ferruginous silica blocks backed by ganister, ordinary silica bricks, and asbestos, was newly installed and had to be dried and preheated for use.

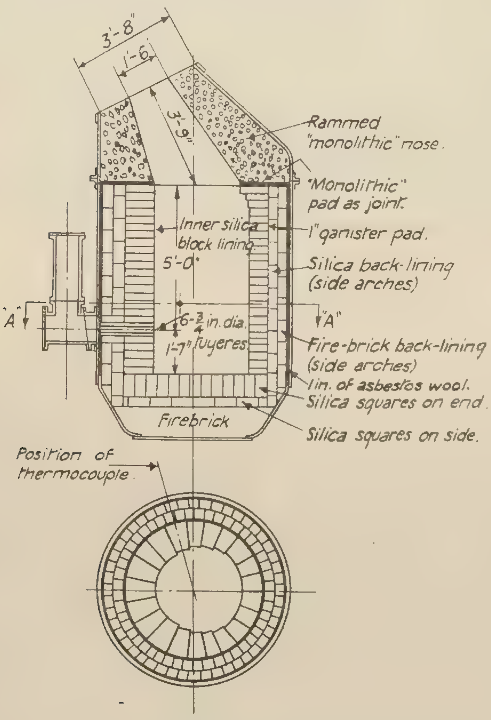


FIG. 2 (*a*)—Details of lining construction (*see* 2 (*b*))

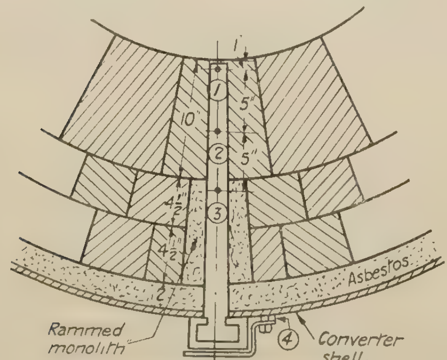


FIG. 2(*b*)—Relative position of thermocouples at "A–A"

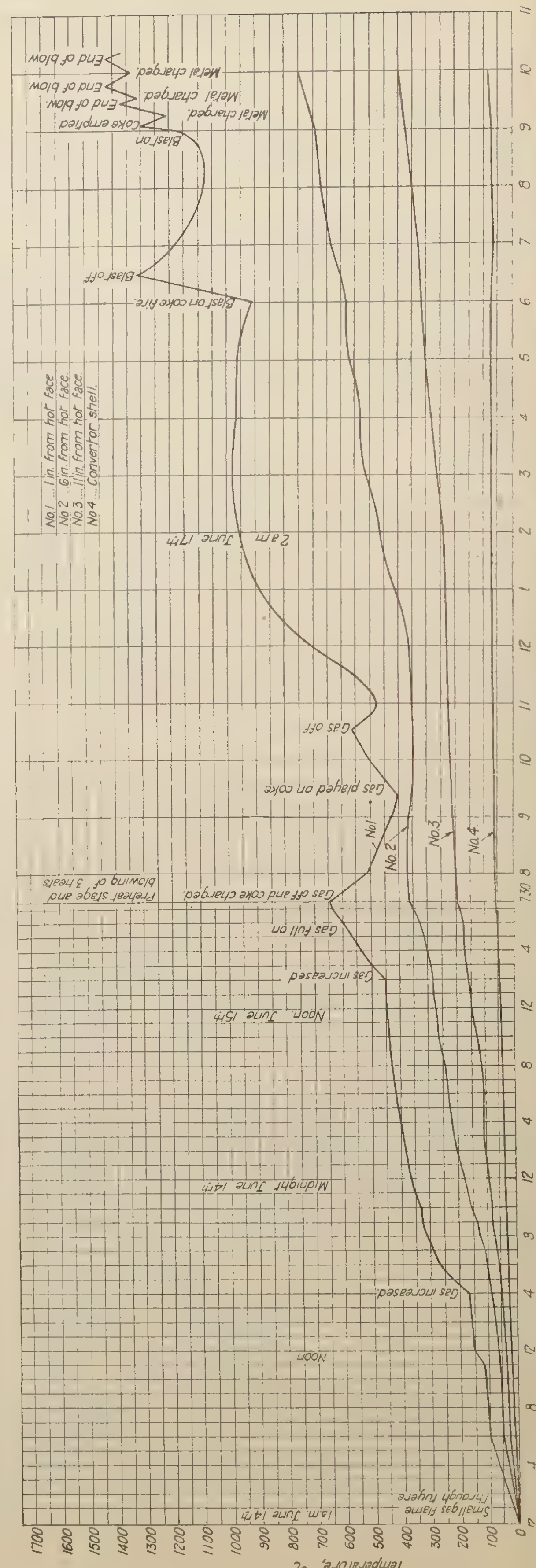


FIG. 2 (*c*)—Temperature curves of silica-brick converter lining during drying, at Plant *C*

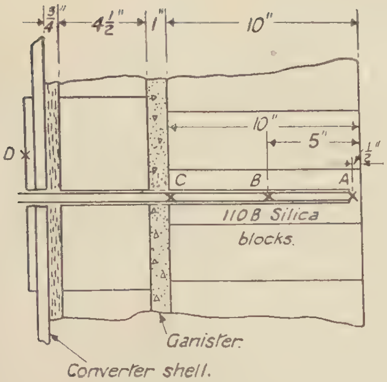


FIG. 3—A diagram showing the position of the thermocouples in the converter lining of Plant T

Drying occupied 71 hr. and the temperatures attained at the end of this period were:

Couple Position	Temperature, °C.
$\frac{1}{2}$ in. from hot face	214
5 ,, ,, ,, ,,	145
10 ,, ,, ,, ,,	95
17 ,, ,, ,, ,,	42

The recorder was then disconnected, the couples removed, and the vessel transferred to the trunnions for preheating and use. The couples were replaced, but a gap of 3 hr. in the records resulted, owing to various delays unconnected with the tests.

Wood was thrown into the vessel and was fired by a small amount of burning coke, and coke was charged on top. The fire was allowed to burn up by natural draught *via* the open tuyeres (blast-box cover off), and the couple at $\frac{1}{2}$ in. indicated a rise from 200° to 680° C. in 2 hr., which, though undoubtedly excessive, gave rise to no spalling as far as could be ascertained. Whether a normal type of silica-brick lining containing more cristobalite and less tridymite would have spalled under these conditions is not known, but it may be significant that the temperature range during which rapid heating occurred is one which excludes the low tridymite inversions, the blocks containing some 70% of tridymite.

The preheating period occupied a total time of 91 hr., due to plant requirements at the time, but this prolonged heating also aided observations by providing several periods when the temperature distribution in the lining was practically static.

Maximum temperature was recorded as 1275° C. by the couple at $\frac{1}{2}$ in. after 62 hr. with the coke fire, using natural draught only. The temperature distribution then was:

Couple Position	Temperature, °C.
$\frac{1}{2}$ in. from hot face	1275
5 ,, ,, ,, ,,	950
10 ,, ,, ,, ,,	660
17 ,, ,, ,, ,,	150

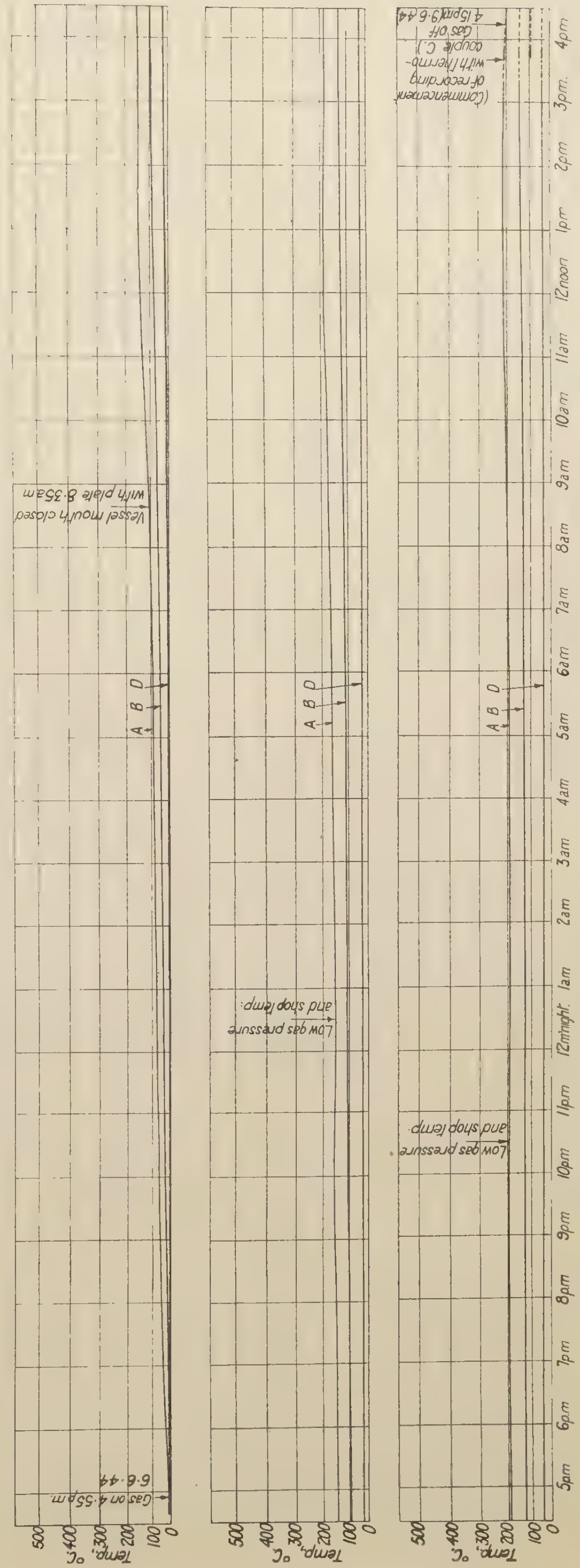


FIG. 4—A reproduction of a chart recording the thermocouple temperatures during drying, at Plant T

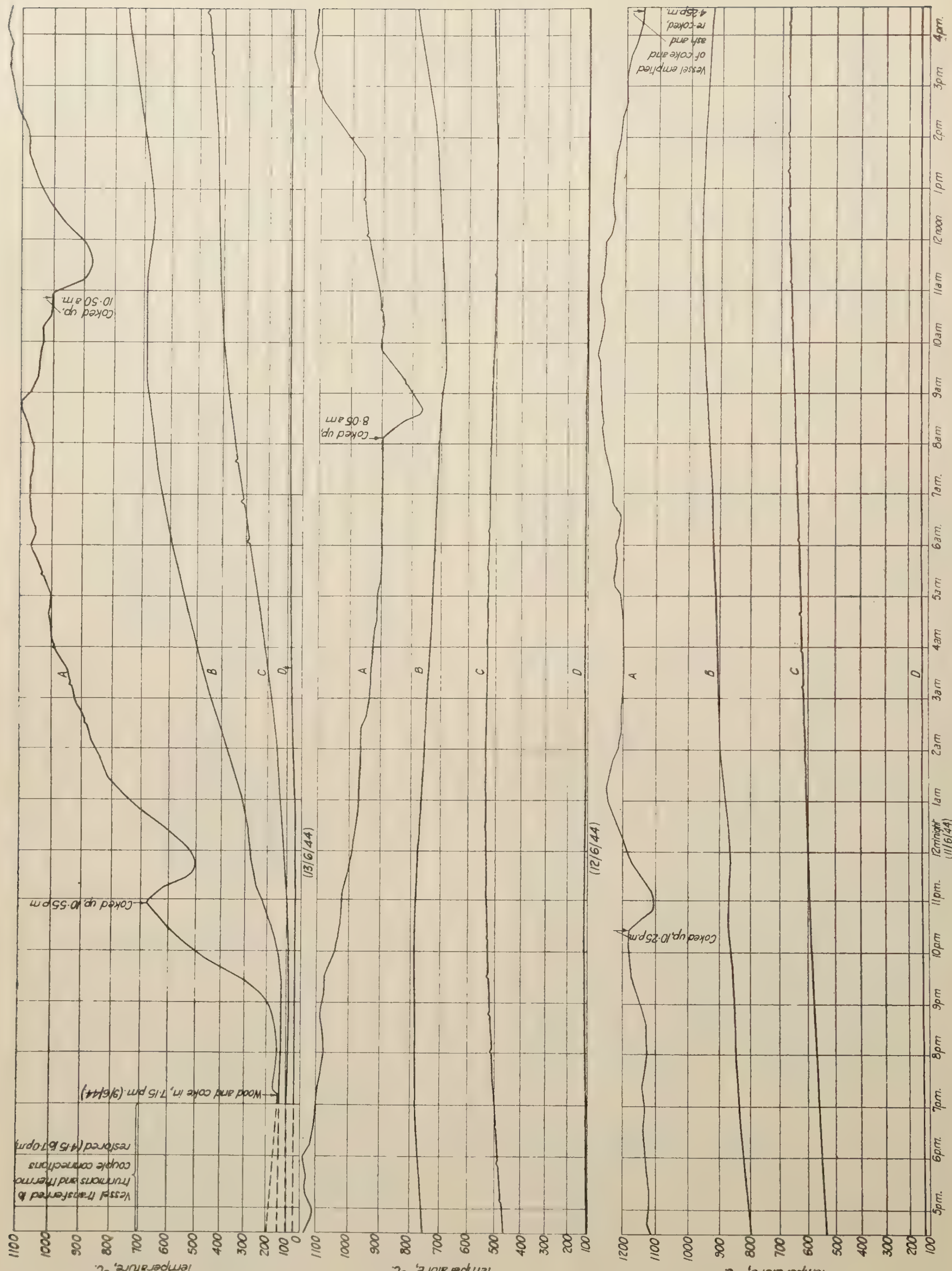


FIG. 5—Temperatures during preheating (continuation of chart in Fig. 4)

TABLE X—*Heating Rates through Inversion Temperatures—Plant* C

Period	Inversion	Temperature, °C.	Temperature Range Selected, °C.	Heating Rate, °C./hr. Couple Position from Hot Face			
				1 in.	6 in.	11 in.	21 in.
Drying (α–β_1) ...	Tridymite ...	117	100–134	6·2	11·3	3·4	Not attained
Drying (β_1–β_2) ...	,, ...	163	146–180	7·6	12·4	5·2	,,
Preheating (	Cristobalite ...	240–275	240–275	17·5	5·2	15·6	,,

A similar, rather lower temperature distribution was attained only 14 hr. after starting the coke fire, thus:

Couple Position	Temperature, °C.
½ in. from hot face	1100
5 ,, ,, ,, ,,	680
10 ,, ,, ,, ,,	380
17 ,, ,, ,, ,,	100

This would have offered quite a suitable occasion for turning on the air blast if the vessel had been required for use at short notice. When the vessel was put into service the application of blast on the coke bed raised the lining temperature ½ in. behind the face from 1180° to 1350° C. in 20 min., *i.e.*, 85° C./min. After 14 heats had been blown and the thickness of the refractory had become reduced by about 0·4 in., the temperature under otherwise similar conditions, rose from 1036° to 1525° C. in 27 min., *i.e.*, 18° C./min. Between the emptying of the vessel of coke and charging the first heat, the temperature fell 85° (8·5° C./min.) and 250° C. (25° C./min.), respectively, in the two cases mentioned above.

Figure 4 is a reproduction of the record of the drying period, and shows the end of the drying period and transfer of the vessel to trunnions. Figure 5 shows the early preheating with coke, whilst Fig. 6 shows the later stages of preheating and the blowing of the first two heats. To spread out the records of the short-duration blows typical of this plant, the chart speed was increased to three times that used for the drying and preheating records.

The rates of heating through the inversion points of the silica allotropes are shown in Tables X and XI for Plants *C* and *T* respectively.

Conclusions and Recommendations Regarding Drying and Preheating

Provided the vessel lining is not allowed to cool below a red heat between successive runs, the following conclusions and recommendations apply only to the initial drying and preheating periods. On all other occasions of preheating the vessel for blowing, the rate of rise of temperature is not critical, as it is unlikely to give rise to spalling owing to the small expansion of silica refractories over the temperature ranges involved.

(*a*) *Drying*

Drying data are only available for Plants *C* and *T*, and lead to the following conclusions:

(1) The drying time (26 hr.) at Plant *C* is satisfactory, but with the gas flare used it might be reduced without danger to the lining, to about 18 hr. This is based on the assumption that drying may be regarded as complete when a temperature of 100° C. is attained at the cooler

TABLE XI—*Heating Rates through Inversion Temperatures—Plant* T

Period	Inversion	Temperature, °C.	Temperature Range Selected, °C.	Heating Rate, °C./hr. Couple Position from Hot Face			
				½ in.	5 in.	10 in.	17 in.
Drying (α–β_1)	Tridymite ...	117	100–134	11·3	2·1	Not attained	Not attained
Drying (β_1–β_2)	,, ...	163	146–180	2·1	Not attained	,,	,,
Preheating (α–β_1) ...	Tridymite ...	117	100–146	(*See* text)	(*See* text)	22·6	1·0
Preheating (β_1–β_2) ...	,, ...	163	146–180	,,	68	19·5	Not attained
Preheating (α–β) ...	Cristobalite ...	240–275	240–275	226	50	34	,,

end of the block constituting the working lining (the couple 11 in. from the hot face).

The drying time (71 hr.) at plant T is unnecessarily long and might be reduced to about 15 hr. without endangering the lining.

(2) With the silica-brick lining employed at Plant C a higher heat input from the gas flare in the first few hours of drying would result in still more gradual heating through the cristobalite inversion range in the hotter regions of the lining.

(3) The long drying time at Plant T is due to insufficient heat input from the gas flare and the maximum resulting temperature is barely sufficient for adequate drying. A larger gas flare (or several smaller flares which could be added one at a time as drying proceeds) would enable both drying and initial preheating (up to 800° or 900° C.) to be carried out efficiently.

(4) Although the rates of temperature rise through the critical ranges are much higher than those recommended for the heating of open-hearth-furnace roofs, and are lower than those found in some converter plants, they gave rise to no perceptible spalling.

(5) A uniform rate of rise of temperature of 10°/hr. from cold to 400° C. at the hot face is considered safe as regards freedom from spalling, and should ensure that the hotter half of the lining block (about 5 in. of the total lining thickness) is above the α–β cristobalite inversion range. With suitable previous arrangements and plant layout, preheating would follow as an automatic extension of the initial drying period.

(b) Preheating

(1) Preheating following initial drying as defined in 'Drying,' conclusion (1), requires careful control if excessive rates of heating are to be avoided. The building of wood, coal, or coke fires in vessels where the average temperature of the working lining is below 250° C. is deprecated, though it is recognized that in many steel foundry plants other facilities may not be available. This calls for a minimum hot-face temperature of 400° C. and a cool-face temperature of 100° C. for a 10-in. block. These temperatures are readily attained by means of suitable gas flares with adequate control of heating rates.

(2) Where it is not practicable to carry out both drying and preheating with the vessel set in the trunnions, initial heating to the temperatures mentioned above will help to ensure freedom from spalling during the transfer of the vessel and resumption of heating.

(3) The heating rates at Plant C are probably satisfactory as regards safety of the brickwork in

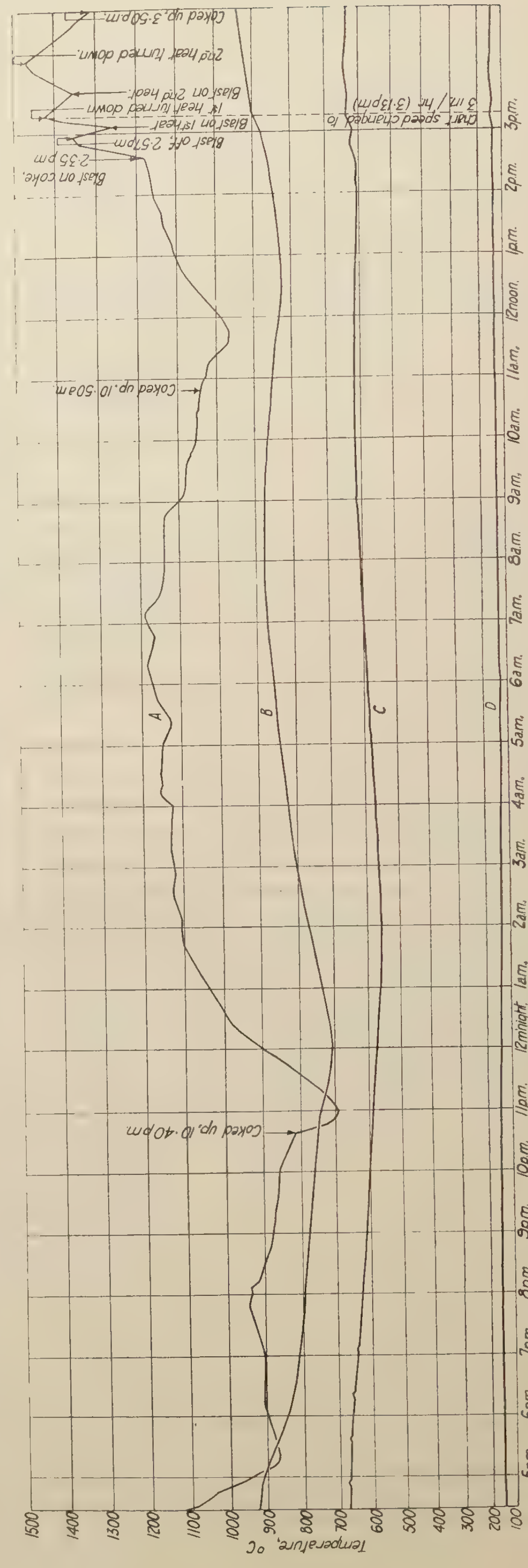


FIG. 6—The later stages of preheating, and the blowing of 2 heats (continuation of chart in Fig. 5)

passing through the critical ranges, but they might well be increased rather than lowered at still higher temperatures, and so save time in preheating. The heating rates at Plant *T* are generally excessive for normal-type silica bricks, though they may be satisfactory for the ferruginous high-tridymite type.

(4) The recommendations made under 'Drying' would obviate much of the danger to be associated with coke-fired preheating, since the coke would only be introduced when the vessel lining had reached 500° C. or higher, up to perhaps 800° to 900° C.

(5) Gas flares for drying and initial preheating are strongly recommended for use where local conditions permit their employment.

Lining Temperature during Blowing

Plant C—Records of the blowing of a few heats were obtained before an accident destroyed the thermocouple installation. It will be seen from Fig. 2 (*c*) that the maximum temperature attained 1 in. behind the hot face rose sharply during the first two heats and then tended to become stable around 1420° C.

Plant T—The couple installation was better protected by its situation than in the previous case, and enabled complete temperature records of the lining throughout 17 heats to be obtained before wear of the lining caused failure of the hot-face couple (at ½ in. depth). Table XII gives the salient details obtained from the records, parts of which, relating to Heats 3 to 14 inclusive, are shown in Fig. 7. Figure 8 shows the portion of the record from Heat 15, through the failure of the hot-face couple in heat 18, to the end of the 28th heat. For convenience, the recorder chart speed was reduced from 3 in./hr. to 1 in./hr. at the beginning of the 15th heat.

From Table XII and Figs. 7 and 8 the following points will be observed:

(i) When blowing the vessel alternately with another vessel, *i.e.*, averaging 9–10 min. blowing and 19–20 min. standing, the excursion of temperature of the three Couples *A*, *C*, and *D* per heat was approximately 200°, 30°, and 15°C., respectively. The maximum temperature shown by Couple *A* (½ in. deep initially) was around 1550° C. when steady cycling had been attained after 4 or 5 heats, and the average temperature was 1450° C.

(ii) Blowing the vessel alone in successive heats, *i.e.*, averaging 8–9 min. blowing and 8–9 min. standing, caused the excursion of temperature to fall to approximately 150°, 20°, and 12° C. for the Couples *A*, *C*, and *D*, respectively. At the same time the average

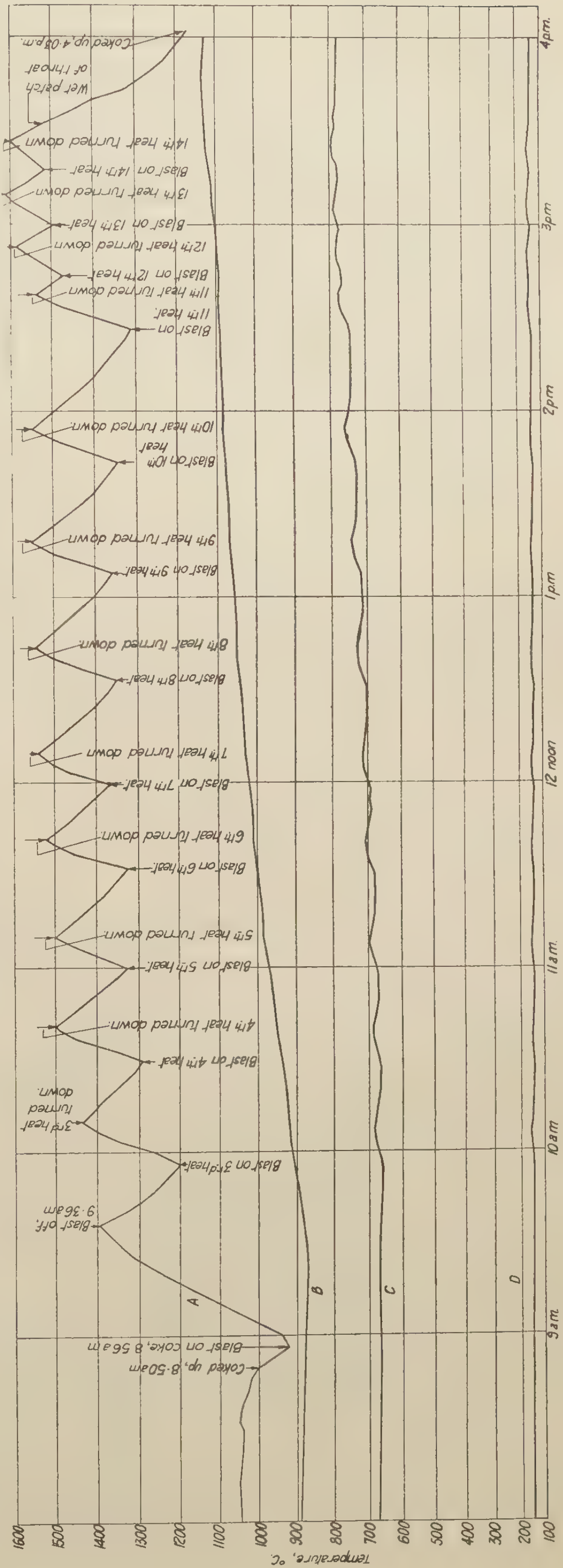


Fig. 7—Continuation of the chart, showing lining temperature fluctuations during Heats 3 to 14 at Plant *T*

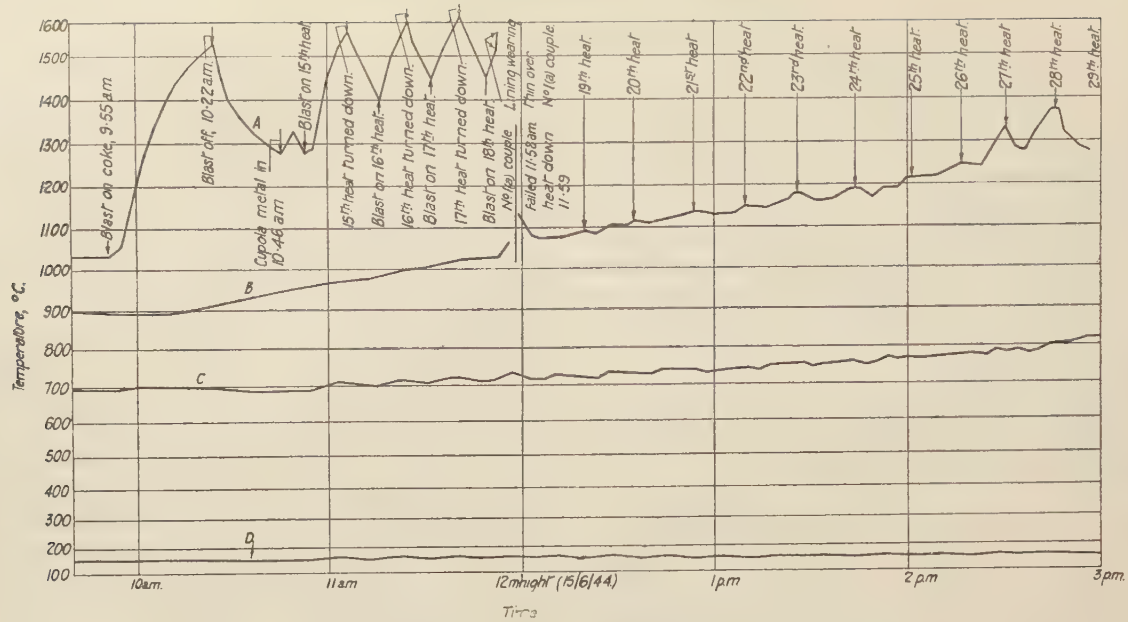


FIG. 8—Fluctuations in lining temperatures during Heats 15 to 28

lining temperature increased to about 1540° C. (Couple *A*) and the maximum was practically 1600° C. The average temperature of Couple *A* was thus some 90° C. higher with sequential blowing than with alternate blowing of the vessel.

(iii) The increase in temperature of the lining due to blowing is clearly discernible in the records of Couples *A*, *C*, and *D*, but less observable in the records of Couple *B*. An explanation of this cannot yet be offered.

(iv) Although fluctuations of temperature due to the blowing of heats are perceptible even in the shell temperature some 17 in. away from the inner face of the brickwork, the variations shown by the different couples do not reach their maxima and minima together. Broadly, the variations are isochronal, but show phase differences.

It would be expected, owing to the relatively low thermal diffusivity of the lining, that Couples

TABLE XII—*Temperature Variations through the Lining during Blowing* (*Plant* T)

Heat	Blowing Time, min.	Interval between Heats, min.	Couple *A* (at ½ in. deep), °C.				Couple *B* (at 5 in. deep), °C.				Couple *C* (at 10 in. deep), °C.				Couple *D* (at 17 in. deep), °C.			
			Min.	Max.	Range	Average	Min.	Max.	Range	Average	Min.	Max.	Range	Average.	Min.	Max.	Range	Average
1	11	8	1263	1422	159	1343	Variations not well defined			...	618	635	17	626	144	155	11	149
2	10		1354	1464	110	1409				...	633	646	13	639	146	156	10	151
3	14	20	1200	1436	236	1318	Variations not well defined			...	663	689	26	676	160	175	15	167
4	10	19	1290	1500	210	1395				...	670	693	23	681	160	172	12	166
5	10	21	1330	1505	175	1417				...	678	700	22	689	161	175	14	168
6	9	18	1327	1523	196	1425				...	684	715	31	699	162	174	12	168
7	9	20	1365	1543	178	1454				...	697	723	26	710	162	170	8	166
8	9	19	1353	1547	194	1450				...	706	736	30	721	160	174	14	167
9	10	19	1355	1558	203	1456				...	720	750	30	735	160	175	15	167
10	10	31	1347	1550	203	1448				...	734	766	32	750	162	179	17	170
11	10	8	1312	1540	228	1426				...	746	780	34	763	165	179	14	172
12	8	7	1470	1581	111	1525				...	770	788	18	779	166	179	13	172
13	9	8	1495	1612	117	1553				...	775	793	18	784	167	180	13	173
14	9		1510	1600	90	1555				...	780	798	18	789	168	181	13	174
15	11	11	1275	1557	282	1416	955	975	20	965	688	714	26	701	160	170	10	165
16	8	8	1400	1580	180	1490	985	1004	19	994	700	720	20	710	160	174	14	167
17	8	9	1433	1590	157	1511	1010	1028	18	1019	710	727	17	718	160	172	12	166
18	9	6	1448	(couple failed 1 min. from end of blow)			1032	1095	63	1063	714	740	26	727	162	170	8	166
19	9	8					1077	1096	19	1086	721	732	11	726	164	172	8	168
20	9	8					1088	1114	26	1101	734	746	12	740	162	172	10	167
21	10	8					1110	1142	32	1126	735	750	15	742	160	172	12	166
22	8	8					1130	1152	22	1141	741	760	19	750	162	170	8	166
23	9	8					1145	1178	33	1161	747	765	18	756	160	170	10	165
24	9	8					1161	1192	31	1176	753	776	23	764	162	172	10	167
25	9	10					1170	1220	50	1197	770	785	15	777	160	168	8	164
26	10	7					1218	1253	35	1235	778	794	16	786	162	170	8	166
27	9	6					1244	1332	88	1288	784	808	24	796	162	170	8	166
28	10						1278	1380	102	1329	808	826	18	817	164	172	8	168

A, *C*, and *D* would reach their maxima at varying periods of time *after* Couple *A*. Whilst this is observed for Couple *C* in Heats 9, 10, and 11, the lag amounting to 0·7 min., it is by no means generally the case. In other heats, Couple *C* reaches its maximum up to 2·5 min. before Couple *A*, whilst Couple *D* records a maximum before the end of the blow in all cases examined. It is not possible to indicate the time lead of Couple *D* with any accuracy, owing to the small temperature excursion, and Couple *B* also fails to help in this respect.

A possible explanation of the observations attributes the effect to the permeability of the lining and the common, but not invariable, reduction of blast pressure at some stage of the blow—generally the carbon boil. No data affording correlation were available and this explanation can only be regarded as tentative.

Metal Temperatures

Table XIII below, shows the blown-metal temperature obtained by the quick-immersion pyrometer with portable potentiometer, for three of the heats, and lists alongside, the corresponding lining temperatures.

TABLE XIII—*Metal and Lining Temperatures* (*Plant* T)

Heat	Temperature, °C.	Lining Temperature, °C.			
		Couples			
		A	*B*	*C*	*D*
1	1678	1422	900	635	155
2	1702	1464	925	646	156
5	1744	1505	988	700	175

Points ½ in. into the lining from the hot face may thus be 240°–250° C. below the temperature of the metal in the well. This observation and the relatively short duration of the blow probably account for the successful use of refractory materials, the ultimate refractoriness of which may be appreciably lower than the temperature of the blown metal.

Sect. B—Study of Silica-Brick Linings

A detailed study of a silica-brick converter lining installed at Plant *C* was undertaken with the object of determining the changes of both chemical and physical characteristics which occurred as a result of service in the vessel. The drying and preheating studies described in the previous section were made with this same lining.

The lining proper was constructed from four different sizes of silica blocks, and the tuyeres, also of silica brick, were made by a different manufacturer from the one who supplied the blocks used in this study. The nose was lined with a rammed monolith. The converter was operated in normal production schedules, but without patching of the body lining in any way. Minor repairs were carried out to the nose–body junction as necessary. About 80 lb. of sand were thrown into the vessel before each metal charge, this being standard practice at the plant in question. After 141 heats had been blown, each of 60–75 cwt., the lining was considered unserviceable and became available for sampling. Figure 9 shows the construction of the vessel and also indicates the amount and position of wear at the end of the run.

SAMPLES

(1) *Before Use*

Representative samples of each of the four sizes of blocks and of the tuyere bricks were taken from the works' stocks at the time the lining was being installed. Two blocks of each size (marked *G*1, *G*2, *G*3, and *G*4) and three complete tuyeres (1¾-in. dia. hole) were selected for laboratory testing.

(2) *After Use*

At the conclusion of the service life of the lining the vessel nose was removed, the lining photographed, and certain areas marked to be reserved as samples. Measurements were also taken which enabled the wear to be indicated on a drawing of the cross-section of the vessel. Figures 10 and 11 show the appearance of the lining after use and indicates the nature and extent of the wear. Details of the position and size of the samples taken after the run are shown in Table XV. Owing to extensive disintegration during cooling and removal, the tuyere sample (*E*) carried none of its original slagged face and was discarded as being of little immediate interest.

TEST PROGRAMME AND RECORDS

The bricks, both used and unused, have been submitted to laboratory examination with regard to chemical composition, mineralogical constitution by X-ray and petrological methods, refractoriness, after-expansion, refractoriness-under-load, bulk density, porosity, and true specific gravity.

Details of the steels made were available from the plant records, and a series of slag samples taken during the life of the lining has been subjected to chemical analysis, X-ray and petrological examination.

Results

The bricks employed in lining the converter were found to have properties before use as shown in Table XIV.

The block samples removed from various parts of the lining at the end of the converter campaign were sectioned so as to expose the zoning which had developed in service, and representative sections from each sample area were photographed in natural colour for record purposes. Typical sections are shown in Figs. 12, 13, and 14.

Representative samples from each of the Areas *A* to *G* (omitting *E*) were further sectioned according to the zones observed, totalling 24 in all. Table XV shows the number and dimensions of the sections in which the blocks were cut, the dimension shown being that in the direction of the lining thickness. The original length of the blocks was 10 in. In some cases the sectioning was determined partly by estimation of possible changes of properties which were not made evident by changes of colour or texture.

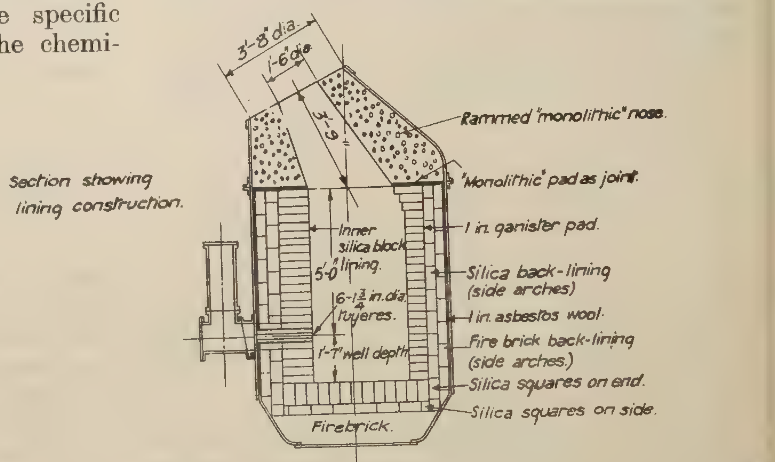
Table XVI shows the results obtained in tests of bulk density, porosity, and true specific gravity, whilst Table XVII records the chemical analyses of the various zones.

The mineralogical constitution of these zoned bricks has also been studied by X-ray crystallographic and petrological methods. The results of the X-ray examination are given in Table XVIII which shows the relative amounts of the different crystalline phases present in each of the zones and in three of the samples of unused bricks. Further subdivision of the hot-face zones was carried out in each case, and the mineralogical changes along a falling temperature gradient thereby rendered more apparent.

Petrological examination of the unused tuyere brick and of

Schedule of Samples from Converter lining after approximately 150 *Heats*

Sample	Position	Details
A	Area including hole for couple, *i.e.*, L.H. trunnion side facing blast-box.	3 courses (centre one containing drilled brick) each comprising equivalent of 2 blocks.
B	As sample *A*, but R.H. side.	As *A*.
C	Blowing side, 4–7 courses above tuyeres.	3 blocks total, 1 block wide.
D	Teeming side, 6–9 courses above tuyere level, slightly to R.H. side of centre.	Area of fairly slag-free brick in region of maximum wear on the teeming side.
E	All tuyeres (preferably whole)	6 tuyeres or all pieces comprising these.
F	Well (middle), blowing side.	Slightly to L.H. side of centre of tuyeres.
G	Well (middle), teeming side.	Slightly to L.H. side of centre.



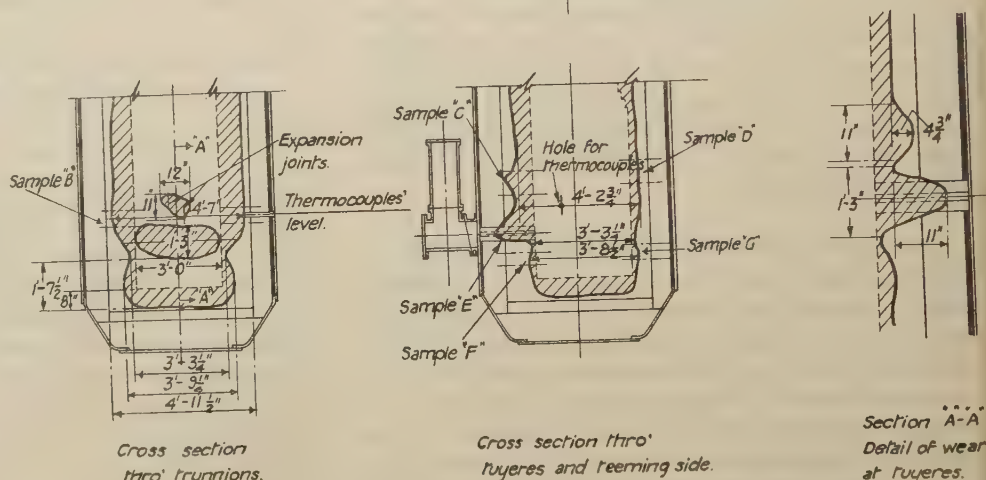


Fig. 9—Sections showing wear of inner lining after 141 heats (Plant *C*)

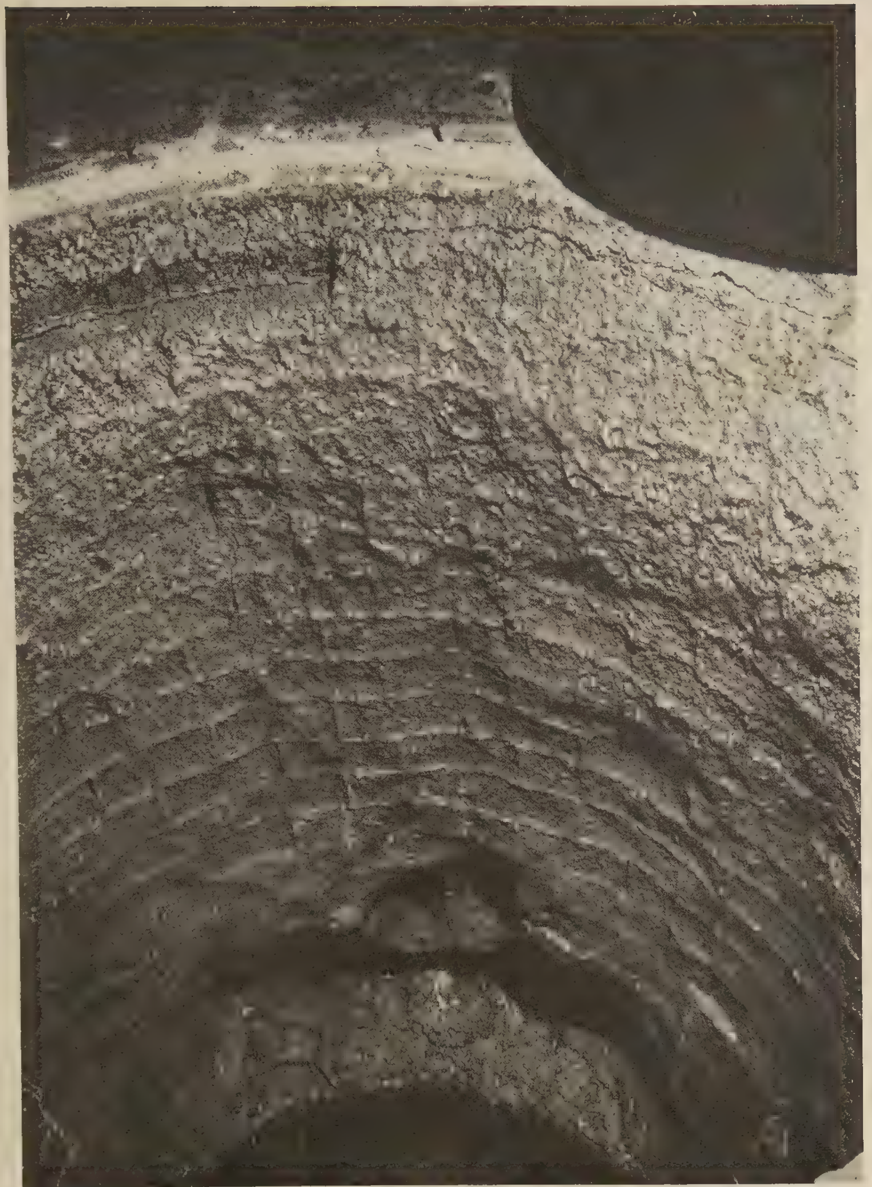


Fig. 10—View of the lining after use (blowing side)

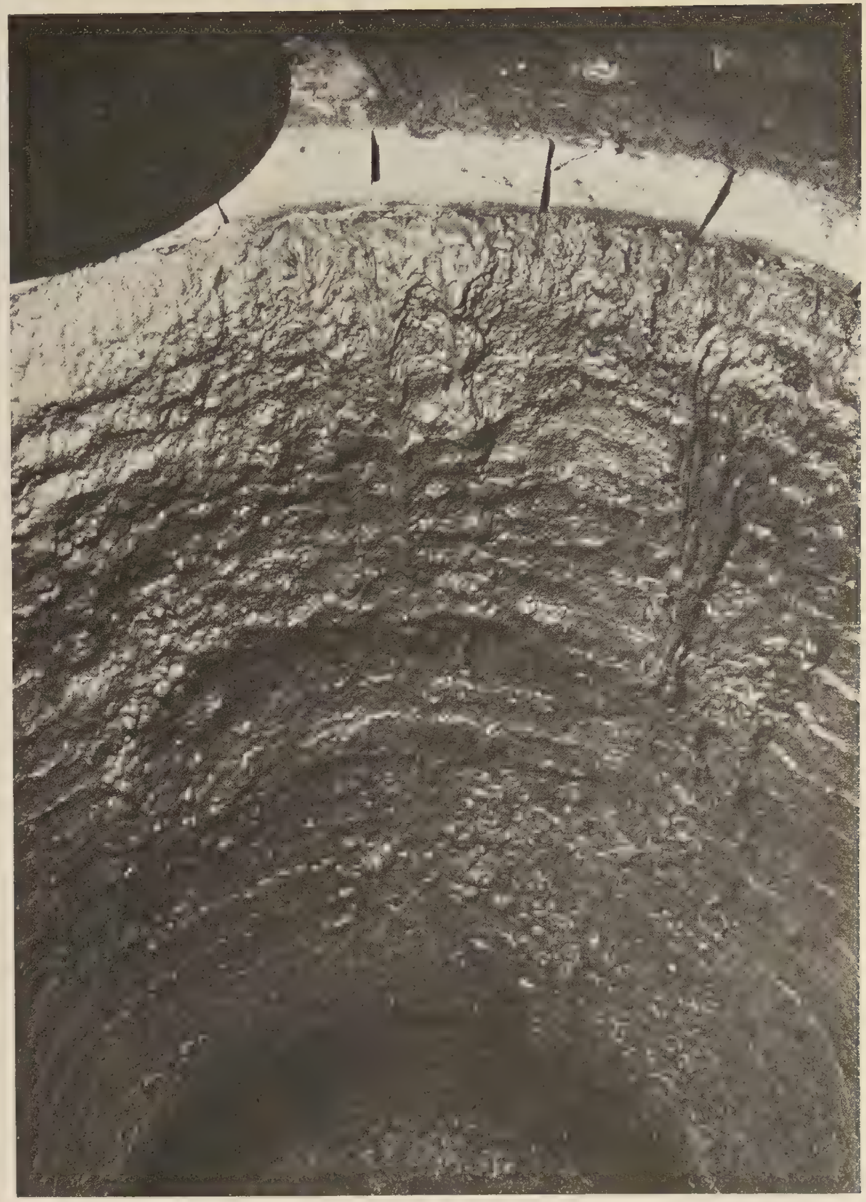


Fig. 11—View of the lining after use (teeming side)

FIG. 12—Sample from Area *A* (left-hand trunnion side facing blast-box, 3–6 courses above tuyeres)

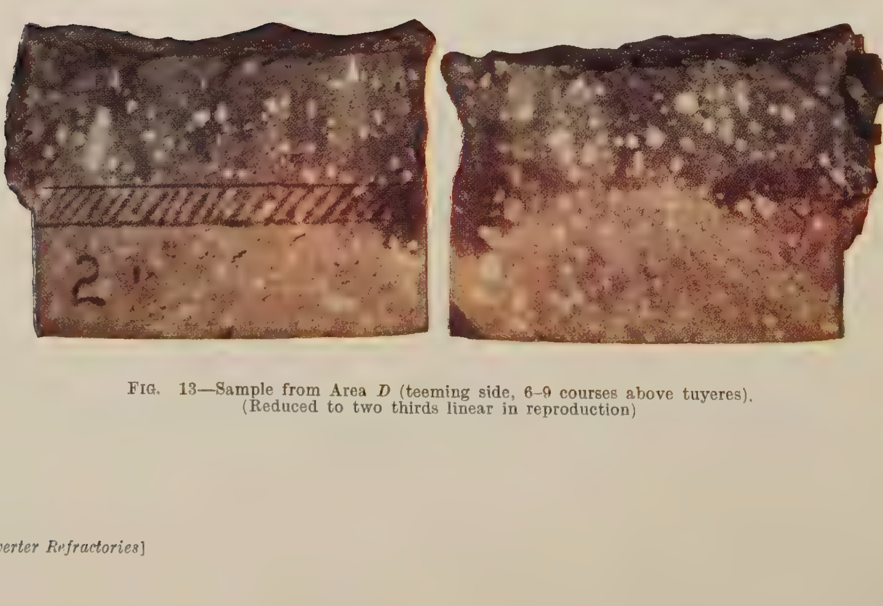


FIG. 13—Sample from Area *D* (teeming side, 6–9 courses above tuyeres). (Reduced to two thirds linear in reproduction)

FIG. 14—Sample from Area *G* (vessel well, 2–3 courses below tuyeres)

TABLE XIV—*Properties of the Bricks before Use*

Properties	Lining Blocks				Tuyere
	G1	*G2*	*G3*	*G4*	*T*
(i) Bulk density, g./ml.	1·71	1·69	1·65	1·68	1·83
(ii) Apparent Porosity, %	27·0	27·5	29·2	27·2	23·10
(iii) True Specific Gravity	2·34	2·3	2·34	2·33	2·38
(iva) Refractoriness (oxidizing atmosphere), °C.	1710	1710	1710	1710	1710
(ivb) Refractoriness (reducing atmosphere), °C.	1680–1725	1685–1725	1685–1725	1680–1725	1675–1725
(v) Refractoriness-under-load (28 lb./sq. in.), 4-hr. maintained temp. test) :					
Subsidence at 1600° C., %	0·92	0·56	0·59	0·74	0·06
Subsidence at 1630° C., %	2·37	0·72	0·76	3·25	0·54
(vi) Chemical analysis :					
SiO_2, %	95·93				95·19
Al_2O_3, %	0·97				0·61
FeO and Fe_2O_3, %	0·92				1·18
Cr_2O_3, %	...				...
MnO, %	...				...
TiO_2, %	0·36				0·26
CaO, %	1·53				2·51
MgO, %	0·04				0·14
SO_3, %	...				...
P_2O_5, %	...				...
Ignition loss, %	0·05				0·08
Total, %	99·80				99·97

the Zones *A1*, *A2*, *A4*, *B1*, *B2*, *B4*, *C1*, *D1*, *F1*, *G1*, and *G2* from the used lining has been made and the observations are summarized below. For key to reference letters, *see* Table XV.

Sample T—*Unused Tuyere*

A coarse-grained silica brick with a maximum grain-size of 3–4·5 mm. dia. Large grains contained a high proportion of quartz of fairly uniform crystal size (average 0·1 mm., maximum 0·5 mm.). The fine grains of 0·1 mm. and less, were generally completely converted to cristobalite though some retained quartz nuclei. The glassy matrix tended to deposit crystals of a wollastonite–$FeSiO_3$ solid solution, and the total free quartz appeared to be 15–20%.

Samples from Area A—*Left-hand Trunnion Side*

Zone A1—Large grains near the working face contained no free quartz and the boundaries showed cristobalite changing to tridymite. Glass and iron oxide filled the interstices between the grains, and free quartz first appeared about 6 mm. behind the working face.

Zone A2—This zone resembled the cool end of the block (Zone 4A) very closely.

Zone A4—The largest grains of this typical coarse-grained brick were 4–5 mm. across. Quartz, extensively cracked, still persisted at the centres of grains though the peripheries showed appreciable cristobalite, whilst the smaller grains (0·15 mm. and less) were entirely cristobalite. The free quartz content was 5–10%, and the yellow-brown glass phase showed some crystallization of tridymite.

Samples from Area B—*Right-hand Trunnion Side*

Zone B2—Five separate zones were distinguished in this section. Progressing inwards from the working face, these were :

(i) A layer of yellow-brown glass, 0·04 mm. thick.

(ii) A zone varying from 0·3 to 1·0 mm. thick consisting of magnetite and recrystallized cristobalite. The latter, the stable phase in this zone, resembled the cristobalite found at the working face of open-hearth-roof bricks and was of larger crystal size than that of unused silica bricks.

(iii) A zone 4 mm. thick comprised cristobalite which had not recrystallized, but contained no free quartz. Cristobalite areas up to

2·5 mm. across were derived from large grains in the block and between them the matrix consisted of tridymite, magnetite, and much glass. The magnetite occurred as dendrites and the glass tended to deposit needles belonging to the triclinic series of solid solutions between wollastonite ($CaO.SiO_2$) and ferrous silicate ($FeO.SiO_2$), and also some tridymite.

TABLE XV—*Details of Used Bricks Examined*

Sample Area	Zone	Thickness in.	Residual Thickness of Brick. in.
A Left-Hand, Trunnion Side (facing blast - box) 3–6 courses above tuyeres.	*A1*	1·6	6·2
	A2	1·4	
	A3	1·4	
	A4	0·8	
B Right-Hand, Trunnion Side (facing blast-box) 3–6 courses above tuyeres	*B1*	1·3	4·9
	B2	1·0	
	B3	1·1	
	B4	−0·5	
C Blowing Side 4–7 courses above tuyeres	*C1*	1·0	5·2
	C2	1·4	
	C3	1·4	
	C4	0·4	
D Teeming Side 6–9 courses above tuyeres	*D1*	1·0	2·3
	D2	0·9	
F Vessel Well, Blowing Side, 2–3 courses below tuyeres	*F1*	2·7	9·3
	F2	2·3	
	F3	1·1	
	F4	0·8	
	F5	1·2	
G Vessel Well, Teeming Side, 2–3 courses below tuyeres	*G1*	1·2	7·0
	G2	1·0	
	G3	1·2	
	G4	1·2	
	G5	1·1	

(iv) A zone 4–12 mm. thick containing little quartz and mainly cristobalite, tending to change to tridymite. About 12 mm. behind the hot face, tridymite was the stable phase.

(v) Between 12 and 25 mm. behind the working face, quartz was present in grains which exceeded 0·1 mm. dia., whilst the pale-yellow-glass areas were free from magnetite.

Zones *B2* and *B4* resembled the corresponding sections from Area *A*.

Samples from Area C—Blowing Side

Zone C1—The hot face comprised black magnetite crystals in a glass varying from deep yellow to a bright orange colour, and recrystallized cristobalite extending to 0·5 mm. from the face. Then well-developed tridymite appeared and quartz was detected 7 mm. behind the working face. Magnetite still appeared in the glass 25 mm. into the block, and seemed more plentiful than in similar samples from Areas *B*, *F*, and *G*, whilst devitrification of the glass was less.

Samples from Area D—Teeming Side

Zone D1—The hot face consisted of a cristobalite layer (max. 0·5 mm.) with some areas of unchanged brick containing tridymite crystals. Further back, a zone in which tridymite was the stable phase occurred, and quartz first appeared some 8 mm. behind the working face. The glass, which had crystallized in parts as wollastonite-ferrous silicate crystals, varied from almost colourless, through pale green, lemon-yellow and brown, to a dirty grey, and was generally greener than that in *B1*.

TABLE XVI—*Physical Properties of Used Silica Bricks*

Zone	Bulk Density, g./ml.	Apparent Porosity, %	True Specific Gravity
A1	2·12	12·1	2·43
A2	1·71	26·4	2·34
A3	1·71	26·8	2·35
A4	1·70	27·1	2·33
B1	2·06	13·5	2·43
B2	1·68	27·7	2·35
B3	1·67	28·0	2·34
B4	1·71	26·6	2·31
C1	2·15	10·8	2·43
C2	1·71	26·2	2·36
C3	1·70	26·8	2·34
C4	1·73	25·4	2·31
D1	2·08	14·0	2·45
D2	1·71	26·6	2·33
F1	2·08	14·3	2·44
F2	1·73	25·6	2·38
F3	1·73	25·6	2·34
F4	1·72	26·1	2·33
F5	1·73	25·3	2·34
G1	2·16	11·8	2·48
G2	2·04	10·8	2·45
G3	1·68	27·8	2·33
G4	1·69	37·6	2·34
G5	1·68	27·8	2·33

Samples from Area F—*Well, Blowing Side*

Zone F1—This section was the most interesting of the series. From the face to 1·4 mm. back, magnetite and recrystallized cristobalite were the only crystalline phases, the glass being yellow–orange in colour. The crystal size of the cristobalite formed from the centres of original grains was fine, whilst the peripheries showed coarse cristobalite which, near the hot face (0·05 mm. away) has recrystallized. Glass, which had deposited magnetite, had penetrated to the centre of the largest grains which consisted of cristobalite with tridymite peripheries. Extensive devitrification of the glass occasionally showed laths up to 0·5 mm. in length, which were recognized by their optical properties as belonging to the triclinic wollastonite–ferrous silicate series of solid solutions. Quartz first appeared at the centre of the largest grains some 8 mm. behind the working face.

Samples from Area G—*Well, Teeming Side*

Zone G1—Like the section from Zone *D1*, some tridymite appeared at the hot face. Apart from much glass, which in a few large areas had crystallized, the rest of the section consisted mainly of recrystallized tridymite. No quartz was seen up to 20 mm. from the working face.

Zone G2—The largest grains (4·8 mm.) consisted mainly of cristobalite with quartz nuclei, and such nuclei persisted in grains of down to 0·2 mm. dia. Tridymite occurred in the matrix.

These observations may be summarized in the following terms:

(1) Changes in the mineralogical constitution of the block arising from service in the converter were confined to the first 2 in. behind the working face.

(2) The first 1 in. of this region shows penetration of a green–yellow glass accompanied by magnetite. The glass persists at a greater distance from the hot face than the magnetite, which appeared as cubic crystals near the face, and as dendrites growing in the glass further into the brick.

(3) For a very short distance, usually about 1 mm. or less, the brick face consisted of recrystallized cristobalite resembling that in the cristobalite zone of an open-hearth-furnace brick, though the latter is commonly 1 in. or more in depth.

TABLE XVII—*Chemical Composition of Used Silica Bricks*

Zone	SiO_2, %	Al_2O_3, %	FeO, %	Fe_2O_3, %	Cr_2O_3, %	MnO, %	TiO_2, %	CaO, %	MgO, %	SO_3, %	P_2O_5, %	Ignition Loss, %	Ignition Gain, % *	Total, %
A1	86·21	1·69	5·00	2·22	0·02	1·93	0·41	2·07	0·12	0·17	0·04	...	0·39	99·88
A2	95·89	0·76	Nil	0·73	Trace	0·02	0·38	1·60	0·08	0·12	0·02	0·14	...	99·74
A4	95·61	0·73	0·40	0·42	Trace	0·06	0·36	1·71	0·04	0·12	0·02	0·21	...	99·68
B1	86·03	2·06	3·60	2·94	0·01	2·43	0·42	2·48	0·16	0·18	0·05	...	0·24	100·36
B2	95·69	1·58	0·20	0·68	Trace	0·02	0·35	1·48	0·07	0·07	0·03	0·11	...	100·28
B3	95·51	1·00	0·10	0·70	Nil	0·03	0·35	1·48	0·05	0·07	0·02	0·11	...	99·42
B4	95·39	0·90	Nil	0·78	Nil	0·02	0·35	1·55	0·02	0·10	0·01	0·10	...	99·22
C1	84·16	1·38	6·60	0·96	0·01	3·42	0·45	2·15	0·13	0·07	0·03	...	0·37	99·36
C4	94·23	1·00	0·30	0·92	...	0·07	0·39	2·09	0·06	0·05	0·02	0·06	...	99·19
D1	85·21	1·73	6·10	2·30	0·01	1·60	0·27	2·35	0·10	0·08	0·03	...	0·71	99·78
D2	95·56	0·75	0·50	0·27	...	0·04	0·41	1·76	0·05	0·04	0·01	0·03	...	99·42
F1	87·23	1·40	4·60	2·93	0·01	0·21	0·62	2·86	0·09	0·08	0·04	...	0·75	100·07
F2	94·25	1·97	0·60	0·37	...	0·02	0·48	1·88	0·10	0·03	0·02	...	0·03	99·72
F3	95·08	0·66	0·60	0·21	...	0·01	0·51	1·79	0·06	0·05	0·01	0·05	...	99·03
F4	95·57	0·63	0·10	0·63	...	Trace	0·52	1·78	0·04	0·07	0·01	0·05	...	99·40
F5	95·31	0·62	0·20	0·63	...	0·01	0·52	1·83	0·05	0·15	0·01	0·20	...	99·53
G1	79·88	2·85	8·50	1·52	0·02	5·01	0·29	1·02	0·18	0·11	0·05	...	0·87	99·43
G2	87·56	2·67	3·30	1·40	...	1·44	0·39	2·47	0·21	0·08	0·03	...	0·38	99·55
G3	96·50	1·20	0·20	0·44	...	0·01	0·29	1·52	0·05	0·05	0·02	0·08	...	100·36
G5	95·61	1·48	0·30	0·51	...	0·04	0·27	1·47	0·07	0·07	0·01	...	0·08	99·83

* Excluded from total

(4) Large grains at the hot face already converted to cristobalite often failed to recrystallize.

(5) From 4 to 12 mm. behind the cristobalite region, tridymite was the stable phase and appeared to have crystallized from the glass, being found in the matrix. In the same region, large cristobalite grains tended to invert to tridymite at the peripheries where in contact with the glass.

(6) Quartz first appeared about 8 mm. behind the working face.

(7) Devitrification of the glass often produced crystals having optical properties corresponding with those of the triclinic wollastonite–ferrous silicate ($CaO.SiO_2$–$FeO.SiO_2$) series of solid solutions.

Discussion of the Results

(i) *Bricks before Use*

The results given in Table XIV and those appertaining to the bricks before use in Table XVIII show that the lining was built from typical silica bricks of good quality. The tuyeres were less converted in the manufacturer's kilns than the blocks (*G1* to *G4*) from a different source, and this is borne out by the X-ray and petrological examinations reported. Refractoriness, whether under oxidizing or reducing conditions, was satisfactory, the lower figure in the latter case

Table XVIII—*X-Ray Examination of Silica Bricks*

Zone	Description	Quartz %	Relative Amounts of Crystalline Phases				
			Cristobalite, %	Tridymite, %	Wollastonite, %	Fayalite, %	Magnetite, %
T	Tuyere, unused	15	35	50	...	...	...
G1	Lining, unused...	<5	45	50	<5	...	...
G2	Lining, unused...	<5	45	50	<5	...	...
A1 (i)	0–1/8 in. slagged surface	...	50	25	...	...	25
A1 (ii)	3/4 in. from surface, black	...	15–20	75	...	...	5–10
A2	...	<5	45	50	<5	...	...
A3	...	<5	45	50	<5	...	...
A4	...	<5	45	50	<5	...	...
B1 (i)	1/8–3/8 in. from surface, dark-grey ...	...	20	70	...	...	10
B1 (ii)	3/4 in. from surface, grey	...	40	50–55	...	...	5–10
B2							
B3 and	...	5	50	45	?	...	...
B4	...	<5	50	45	<5	...	...
C1 (i)	1/16–3/16 in. from surface, dark-grey–black	...	70	15	...	...	15
C1 (ii)	3/4 in. from surface, grey	...	60	40	...	...	...
C2, *C3*, and *C4*	...	<5	40	55	?	...	...
D1 (i)	Slagged surface, black, shiny ...	...	50	...	...	...	50
D1 (ii)	1/4–1/2 in. from surface, dark-grey ...	...	20	50	...	30	...
D2	...	...	35	65	...	...	...
F1 (i)	Slagged surface, black, shiny ...	...	50	...	...	...	50
F1 (ii)	1/4 in. from surface, black	...	20	60	...	20	...
F1 (iii)	1 3/4 in. from surface, dark-grey ...	...	30	70	...	...	...
F2	Grey	<5	50	45	<5	...	...
F3 and							
F4	...	<5	45	50	<5	...	...
F5	...	<5	50	45	?	...	...
G1 (i)	Slagged surface	...	40	...	...	...	60
G1 (ii)	1/2 in. from surface, dark grey ...	...	20	65	...	...	15
G1 (iii)	3/4 in. from surface, brown	...	15	80	...	...	5
G2 (i)	Dark-grey	...	10	75	...	15	...
G2 (ii)	Grey	<5	50	45	<5	...	...
G3	...	5	50	45	?	...	...
G4	...	10	50	35–40	<5	...	...
G5	...	10	50	40	...	...	...

indicating the beginning of deformation of the test cone.

A valuable assessment of the differences between the various bricks emerges in the results of the maintained load and temperature test. Whilst the less converted tuyere block *T* shows negligible subsidence during a 4-hr. test under a load of 28 lb./sq. in. at 1600° C., the others (*G1* to *G4*) show much higher values, varying from 0·56% to 0·92%. On repeating the test at 1630° C., the differences are still more marked and the subsidence increases generally with increased degree of conversion of the brick.

(ii) *Bricks after Use*

These results are considered under :

(*a*) Chemical composition
(*b*) Mineralogy, and
(*c*) Physical properties

(*a*) *Chemical Composition*—Comparison of the analyses of the various zones of the used bricks with those of the bricks before use shows that near the working face considerable increases of FeO, Fe_2O_3, MnO, Al_2O_3, and CaO occur, and smaller increases of TiO_2, MgO, SO_3, and P_2O_5. The silica content of these regions is correspondingly lowered by dilution. The net gains and losses of the constituent oxides are readily seen by calculating the weighted average analyses of the used bricks and comparing these with those of the unused bricks. Table XIX summarizes the comparison in terms of average percentage gain or loss of the various oxides in different parts of the vessel.

The constituents SO_3 and P_2O_5 show relatively small changes and have been omitted from Table XIX. The remainder are now considered in turn, taking silica last, since the associated loss figures are essentially the sum of the gains of other oxides.

Al_2O_3—Generally, the gain of alumina averages 0·3% except on the teeming side of the well, where over 1% increase is found. This latter may be due to the accumulation of alumina formed from aluminium added for deoxidation purposes.

FeO—The highest concentrations of this oxide also occur in the well (teeming side), though similar amounts occur on both the blowing and teeming sides above tuyere level. Relatively severe wear is found in these upper regions, though less occurs in the well.

Fe_2O_3—Although variations occur in different parts of the blocks from the various areas examined, the weighted average is surprisingly constant.

MnO—The average gain of MnO (about 0·8%) is constant in Areas *A*, *B*, and *D*, but is much higher in Area *G* and much lower in Area *F*. The unequal distribution in the well would appear to be due to the effect of ferro-manganese added to the blown metal.

TiO_2—Apart from Area *F*, where a notable concentration occurs, changes are generally small. It appears likely that the samples examined from Area *F* had initially an amount of TiO_2 greater than normal.

CaO—The average gain of CaO is fairly constant but the distribution is variable. The highest surface concentration occurs on the blowing side of the well, whilst on the opposite side the maximum CaO content is found in Zone 2, over 1 in. behind the working face. Drainage of lime-bearing liquid between blows may account for this distribution.

MgO—Small increases of MgO occur in all parts of the vessel and there is some indication of increased concentrations on the teeming side of the well behind the working face.

SiO_2—The dilution of silica is greatest on the teeming side both above and below tuyere level, and the effect, like the gains of other oxides, is unequal on the trunnion sides. It is noteworthy that the concentration of silica at the hot face of the converter lining is at least as low as that found in acid-open-hearth-furnace silica bricks, whilst the nature and distribution of the fluxing oxides is even less favourable to the durability of the lining.

(*b*) *Mineralogy*—A converter lining built of silica blocks appears, after use, to present features resembling those observed in open-hearth-furnace silica bricks, but on a much smaller scale. The

TABLE XIX—*Average Gain or Loss of Oxides during Service*

The bracketed figures are considered doubtful owing to insufficient analytical data

Oxide	Left-Hand Trunnion Side, Area A	Right-Hand Trunnion Side, Area B	Blowing Side, Area C	Teeming Side, Area D	Well, Blowing Side, Area F	Well, Teeming Side Area G
SiO_2, %	−4·2	−3·6	(−9·6)	−5·8	−3·6	−6·1
Al_2O_3, %	+0·2	+0·5	+0·3	+0·3	+0·3	+1·1
FeO, %	+2·1	+1·2	(+4·7)	+3·4	+1·7	+3·0
Fe_2O_3, %	+0·5	+0·6	+0·1	+0·5	+0·5	+0·1
MnO, %	+0·8	+0·8	(+2·5)	+0·8	+0·1	+1·7
TiO_2, %	+0·03	+0·01	+0·08	+0·02	+0·18	+0·05
CaO, %	+0·3	+0·3	+0·6	+0·5	+0·7	+0·1
MgO, %	+0·05	+0·05	+0·06	+0·04	+0·04	+0·09

whole sequence of zones is confined to the hotter 2 in. of the brickwork, compared with 6–12 in. in the open-hearth furnace. This appears to be mainly the result of the steeper temperature gradient in the converter lining and the intermittent nature of the supply of heat and fluxes.

Devitrification of the glass phase appears different from that observed in silica bricks taken from basic open-hearth roofs in that the crystals are solid solutions of wollastonite and ferrous silicate instead of mellorite (mainly ferric oxide and lime silicate with small amounts of ferrous oxide, titania, and alumina), but the distinction may be largely an academic one.

X-ray examination generally indicates preponderance of cristobalite near the working face, except in the samples from the well of the converter where tridymite predominates. Since the brickwork in this region is below the metal level during blowing, the temperature can hardly be lower than that prevailing higher up the vessel. yet the conditions appear favourable to tridymite development. The zone of modification is thicker in the well brickwork than elsewhere, due presumably to the greater cumulative effects of time and temperature.

(c) *Physical Properties*—Near the hot-face of samples from all areas examined, the bulk density and true specific gravity rise sharply, due to pick-up of iron oxide and reduction of porosity. The porosity of these regions is generally less than half that of the unused bricks and comprises both open and closed pores, the latter ranging up to 6% in the well and up to 2% elsewhere. Unaltered portions of the brick show nil to 0·5% of closed pores, and these values are typical of silica bricks generally.

Steel and Slag Records

During the run of the vessel lining, nine slag samples were taken after turning the vessel down at the end of the blow, and these have been analysed and studied by petrological and X-ray methods. Table XX gives the details of slag composition with relevant data on the composition of metal charged, blown metal, details of additions to the vessel before or during the blow, and the blowing time.

Whilst the charge weight was increased from 60 to 75 cwt. as the vessel lining wore thinner, the sand addition was maintained constant at 80 lb. throughout. This sand is added to provide free silica in a fairly fine state of division to combine with the quantities of basic oxides (of iron and manganese) which preponderate in the early stages of the blow. In this way the life of the lining is prolonged considerably at this plant (*C*).

Although there were minor variations of composition of both charge and blown metal, much wider variations were found to occur in the compositions of the blowing slags. In particular, the silica content ranged from 63·2 to 81·3%, in three instances exceeding 70%, whilst the ferrous

Table XX—*Metal and Slag Data for Typical Heats*

Heat	2	13	29	58	61	93	107	123	140
Analysis :									
Wt. of metal Charged, cwt. ...	60	60	60	65	65	70	75	75	...
C, %	2·73	3·05	2·54	3·00	2·92	2·89	2·73	2·98	...
Si, %	0·07	0·15	0·09	0·09	0·09	0·07	0·09	0·12	...
Mn, %	0·39	0·37	0·32	0·30	0·24	0·27	0·34	0·44	...
Analysis of Blown Metal :									
C, %	0·08	0·09	0·05	0·08	0·09	0·09	0·11	0·13	...
Si, %	0·02	0·03	0·04	0·04	0·04	0·04	0·04	0·04	...
Mn, %	0·09	0·08	0·06	0·09	0·06	0·08	0·09	0·07	...
Blowing Time, min.	14	11½	11	15	12½	13	...	...	...
Analysis of Blowing Slag :									
SiO_2, %	70·9	73·9	68·3	81·3	63·2	67·2	63·6	64·1	68·8
Al_2O_3, %	3·3	3·8	3·8	3·8	3·8	3·5	4·0	2·6	3·8
Fe_2O_3, %	6·7	5·4	6·3	1·8	2·3	2·0	5·0	8·0	5·2
FeO, %	14·1	8·9	13·6	5·9	24·9	20·1	18·6	14·1	14·9
MnO, %	5·3	8·1	7·7	4·7	5·3	6·9	8·6	10·6	7·0
CaO, %	0·2	0·2	0·2	0·2	0·2	0·2	0·2	0·2	0·2
Additions :									
Sand, lb.	80	80	80	80	80	80	80	80	...
Ferrosilicon (75%), lb.	30	30	40	10	10	40	10	20	...

oxide content ranged from 5·9% to 24·9%, averaging about 14–15%. Generally, low FeO was found in slags high in SiO_2, suggesting that dilution of a normal converter blowing slag (averaging 56–62% of SiO_2) with excess of silica was occurring. Subsequent tests have shown that the slag from Heat 58 (81·3% of silica) contains 15·5% of undissolved quartz, much of which is recognizable as eroded sand grains. The silica content of the true slag phase is thus 65·8%, a more normal figure. Similar considerations appear to apply in a somewhat less degree to the slags from Heats 2, 13, and 29.

Mineralogy of Blowing Slags

(i) *X-ray Examination*

All samples of slag showed a black skin of varying thickness with inside portions of varying colour and texture. The results are shown in Table XXI, grouped according to the colour of the slag.

All the slags were of the glassy type and fall into four groups depending on the relative abundance of crystalline phases:

(i) Pale green slags containing only traces of a crystalline phase—hedenbergite type (Fe, Mn, Ca)O . SiO_2.

(ii) Grey slags containing small to medium amounts of hedenbergite.

(iii) Dark-grey slags containing fayalite 2 (Fe, Mn)O . SiO_2, and hedenbergite.

(iv) Brown and black slags containing fayalite.

The amount of crystalline substance present increased from 'trace' in the pale-green slags to 'medium' (probably 25 wt.-%) in the brown and black slags. In Table XXII the slags are arranged in order of decreasing total silica content and account is taken of the occurrence of a given slag (depending on the colour and mineralogy of the portion examined) in more than one group.

The presence of the increasing proportions of silica thus tends to change the slag from the fayalite or orthosilicate type (2 RO . SiO_2) to the hedenbergite or metasilicate type (RO . SIO_2), and ultimately to inhibit crystallization under the prevailing conditions of cooling rate of the sample, etc.

The white lumps consisting essentially of silica, observed in many of the samples, may have been derived from the lining (pieces of brick), whilst the porous lumps appear to be detached from agglomerations of sand or other siliceous material which have escaped solution in the slag.

(ii) *Petrological Examination*

Thin sections of the slags from Heats 2, 29, 58, 61, 93, and 140 were examined.

Transparent areas consisted of clear glass, sometimes stained yellow or brown. The glass contained isolated rounded crystals of quartz which had changed to cristobalite at the peripheries. Occasionally dendrites of cristobalite growing radially into the surrounding glass,

TABLE XXI—*X-Ray Examination of Converter Slags*

Colour	Heat	Crystalline Phases	Amount Present
Pale grey-green	2	None (*i.e.*, completely glassy)	...
	13	Hedenbergite type	Trace
	58	Hedenbergite type	Trace
	140	None	...
Grey	29	Hedenbergite type	Medium to small
	123	Hedenbergite type	Small
		Fayalite type	Very small
Dark-grey	93	Fayalite type	Medium to small
	140	Fayalite type	Small
		Hedenbergite type	Small
	107	Fayalite type	Medium to small
Brown	61	Fayalite type	Medium
Black	107	Fayalite type	Medium
White (large lump)	Many samples	Cristobalite (uncombined silica)	Large
White (lump, porous)	...	Quartz	Trace
Black (skin)	...	Magnetite	Small
		Quartz	Very small

Table XXII—*Classification of Slags by Silica Content*

Heat	Total Silica, %	Colours Observed	Group	Crystalline Phases	Amount Present
58	81·3	Pale green	1	Hedenbergite	Trace
13	73·9	Pale green	1	Hedenbergite	Trace
2	70·9	Pale green	1	Completely glassy	...
140	68·8	Pale green	1	Completely glassy	...
		Dark grey	3	Fayalite ... ... Hedenbergite	Small Small
29	68·3	Grey	2	Hedenbergite	Medium to small
93	67·2	Dark grey	3	Fayalite	Medium to small
123	64·1	Grey	2	Hedenbergite ... ... Fayalite	Small Small
107	63·6	Dark grey	3	Fayalite	Medium to small
		Black...	4	Fayalite	Medium
61	63·2	Brown	4	Fayalite	Medium

fringed the quartz grains, whilst other large areas were almost entirely cristobalite surrounded by colourless glass. Dendrites of magnetite occurred in the darker areas of glass, and some beads of metallic iron were seen. Other areas (relatively few) consisted of needle-like crystals of lime–ferrous metasilicates. Fayalite was not readily detected in any of the samples.

Vitrified masses occurring in white- and green-coloured areas of the sections were almost opaque, but generally showed some crystallization of minerals belonging to the wollastonite–ferrous silicate series of solid solutions.

The petrological examination of these slags thus leads to the same general conclusions as those reached by the X-ray study and clearly confirms the existence of undissolved sand grains which chemical analysis has revealed and which the X-ray study indicated.

Conclusions

The following conclusions are based on the results of the experimental work with brick-lined vessels. A study of monolithic linings forms part of the future programme of the Panel.

1. Drying and preheating of brick converter linings may be much more rapid than is generally the present practice, provided suitable precautions are taken to ensure freedom from spalling. Such precautions include the use of gas flares for drying and initial preheating, and control of the rate of temperature rise with the aid of temperature recording instruments.

2. The charging of coke into vessels at low temperature (200°–400° C.) leads to excessive rates of temperature fluctuation in the temperature range where spalling is most likely to occur. By heating the lining to 800°–900° C. by means of gas flares before charging the coke, the resulting temperature changes are rendered innocuous.

3. Blowing of successive charges in the same vessel may raise the average hot-face temperature of the lining about 90° C. above that prevailing when the vessel is blown alternately with another. The actual temperature increase will depend on the interval between blows, and the figure quoted relates to plant conditions involving the minimum of delay between successive heats.

4. Additions of sand to the vessel before charging increase the service life of the lining considerably when blowing low silicon irons. Further study under controlled experimental conditions is necessary before a definite statement can be made of the benefit likely to accrue in other cases.

5. The slags obtained from heats to which sand has been added may contain up to 15% of undissolved sand grains for additions of 80 lb. of sand per ton of iron charged.

6. Vessel slags are principally of the metasilicate or orthosilicate types. With increasing silica content, the fayalite slags tend towards the hedenbergite type, until, with excessive silica contents, crystallization is delayed and glassy slags result.

7. Significant grains of alumina, ferrous oxide, manganese oxide, and lime occur during service in all parts of the lining, though the effect is generally greatest on the teeming side of the vessel. Manganese oxide and alumina are found in largest amounts in the well, probably due to circumstances associated with the deoxidation of the blown metal.

8. Drainage of lime-bearing liquid between blows results in concentration of lime at the surface on the blowing side, but some distance into the lining on the teeming side.

9. The silica content of the lining near the working face is comparable with that observed in acid-open-hearth-furnace brickwork, but the

nature and distribution of the fluxing oxides is less favourable to the durability of the lining.

10. The general sequence of zoning which develops in the lining during service resembles that found in open-hearth-furnace silica bricks, but is confined to a region extending only some 2 in. behind the working face.

11. Whilst cristobalite (as in open-hearth-furnace-roof bricks) preponderates near the working face in regions above the metal level, tridymite development is favoured by the conditions prevailing below metal level.

Summary

The outstanding points of each section of the Report are the large variables in the industry regarding the nature and methods of the use of refractories.

The very high consumption of refractories noted in certain cases is obviously capable of improvement. There are certain anomalies in the characteristics of some monolithic linings which are being used with a measure of success. These facts alone focus attention on the necessity for further work in order to enable recommendations to be more specific with regard to the best materials and technique of use.

It is considered that operational factors have played a large part in deciding the performance of materials and have made it impossible to correlate fully such performance with laboratory data.

Any further work carried out must be on a more comparable basis than exists in the various plants from which data have been obtained, and the first recommendation is that plant should be available for use in future experimental work.

The investigations have made available a certain amount of information which is put forward as a basis for discussion :

(*a*) Large vessels are more economical in consumption of refractories than small ones.

(*b*) Current practice relates the size of the converter to the production of the steel required per day being available from about 15 heats.

(*c*) Monolithic linings are preferred for small vessels, with a gradual change over to silica brick for the largest vessels.

(*d*) It has been established that improvement in lining life follows the use of sand charged into the converter before the metal. This applies particularly to plants using low silicon iron.

(*e*) The silica content of monolithic lining materials should be over 90%, and the clay constituent should be sufficiently fine to develop adequate plasticity when present in relatively small amount.

(*f*) The bricks used for converter linings are generally of the type used in open-hearth-furnace roofs, although a special high iron oxide type with improved thermal shock resistance is specially manufactured for converters. The samples examined had a wide range of specific gravity and therefore will require varying degrees of control on heating up.

(*g*) The relative merits of monolithic and brick linings can only be assessed by carefully controlled experiments in the same plant. Experiments on these lines are visualized in the future programme of the Panel.

Acknowledgments

The Refractories Panel wish to thank the following firms for their contributions to this Report in the form of detailed information regarding their converter operation and lining practice. The last five firms, in addition, kindly supplied samples of monolithic materials for laboratory examination :

Messrs. Edgar Allen & Co., Ltd.
Butterley Co., Ltd.
Carntyne Steel Castings Co., Ltd.
Messrs. Catton & Co., Ltd.
Coltness Iron Co., Ltd.
Messrs. Thomas Firth and John Brown, Ltd.
Messrs. Hadfields, Ltd.
Messrs. Head, Wrightson & Co., Ltd.
Messrs. Kryn & Lahy, Ltd.
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Messrs. Samuel Osbourne & Co., Ltd.
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Stanton Ironworks Co., Ltd.
Vickers-Armstrong, Ltd.
The United Steel Companies, Ltd.
Workington Iron and Steel Co.
Messrs. Richard Thomas and Baldwins, Ltd.
North British Steel Foundry, Ltd.
Messrs. F. H. Lloyd & Co., Ltd.
Thomas Summerson & Sons, Ltd.

Thanks are also due to the Directors of the following firms for facilities for experiments on a practical scale and laboratory investigation of a large range of samples taken during the experiment :

Messrs. Hadfields, Ltd.
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The United Steel Companies, Ltd.
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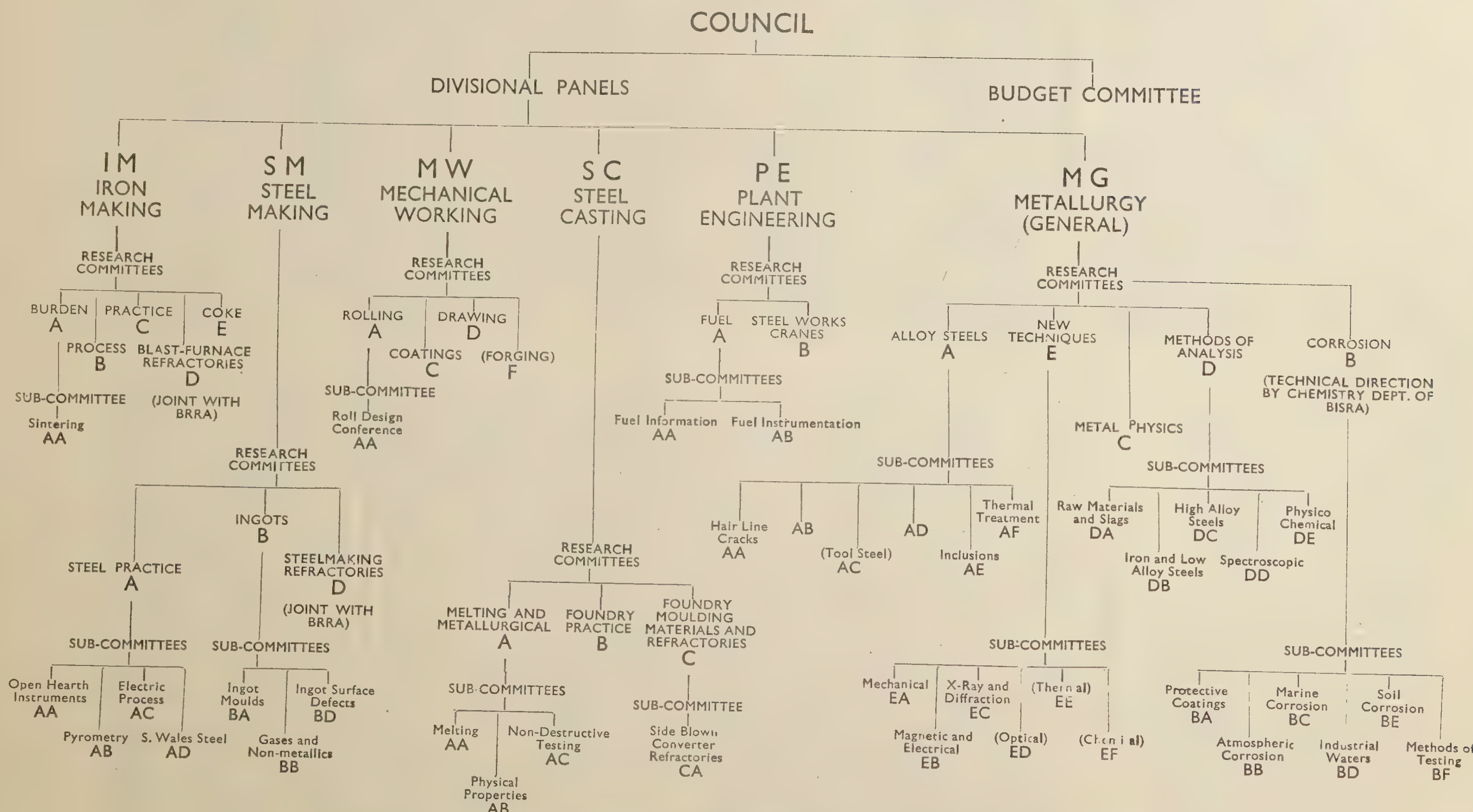
Messrs. Edgar Allen & Co., Ltd.
The British Refractories Research Association.

Finally, the Panel gratefully acknowledge the kind offer of Messrs. Catton & Co., Ltd., Leeds, to conduct further experiments of converter lining materials on a practical scale under comparate conditions.

COMMITTEE ORGANIZATION

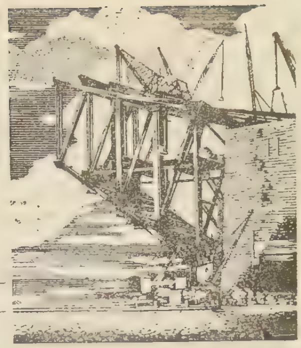
THE BRITISH IRON AND STEEL RESEARCH ASSOCIATION



Where the name of a Committee is placed in brackets, it is proposed but has not yet been formed.

IRON AND STEELWORKS ENGINEERING

THE IRON AND STEEL ENGINEERS GROUP



THE IRON AND STEEL ENGINEERS GROUP

REPORT OF THE FIRST MEETING

THE FIRST MEETING OF THE IRON AND STEEL ENGINEERS GROUP of the Iron and Steel Institute was held at the Offices of the Institute, 4, Grosvenor Gardens, London, S.W.1, on Wednesday, October 16th, 1946. Mr. W. F. CARTWRIGHT occupied the Chair, and Dr. C. H. DESCH, F.R.S., President of the Iron and Steel Institute, was present at the opening of the meeting.

PROCEEDINGS OF THE AFTERNOON SESSION: 2.15 P.M. TO 4.30 P.M.*

DISCUSSION ON A.C. AND D.C. DRIVES FOR STEELWORKS CRANES AND ORE BRIDGES

Mr. L. Rothera (Messrs. Colvilles, Ltd.): I appreciate the honour of being asked to open this discussion. The question of A.C. *versus* D.C. has been discussed ever since A.C. was first developed, which happened to be later than D.C. I am going to advocate D.C. for crane work in steelworks; that is not because I have any grievance against A.C. There is no doubt that if the conditions are known an A.C. crane can be designed to operate perfectly satisfactorily and give you all the movements you need and almost all the control you need, but I am leaving it to other speakers to take up the cudgels for A.C., which they will claim, I expect, to be just as good as D.C. I have not heard anybody claim that it is better.

We use both A.C. and D.C. cranes at our various works, but for heavy duty work I am in favour of D.C., and I have listed the grounds on which I think D.C. is to be preferred.

(1) *Overload Capacity*—This is rather a ticklish point, because there are many crane makers here, and when I say that a D.C. motor will give any overload you like to ask from it, up to five or six times normal, they will probably say that the cranes were not designed for these conditions. From an engineering point of view that is quite correct, but from an operating point of view an overload of that order will very often get you out of difficulty. The modern type of motor which we use, *i.e.*, the mill-type totally enclosed machine, will quite well give four to five times full-load torque momentarily without danger to itself. As against this the mill-type A.C. motor will give only two and a half to three times full-load torque. You may say that overloads of that nature are not wanted for hoists, although they may be required in emergency, but when we come to the question of rapid acceleration on any movement, the overload capacity of the D.C. motor has everything to recommend it.

(2) *Supply Voltage*—In a large works it is rather difficult with long transmission lines and heavy peak loads, to maintain the voltage at the more distant parts of the works, but whereas with the D.C. motor any fall in voltage will only slightly reduce the speed of the machine without affecting its torque, in the case of the A.C. motor the maximum torque is reduced in proportion to the square of the applied voltage. That may be a very serious matter both from the point of view of the motor and from that of the control, because the pull-in coils of the contactor gear may fail to function.

(3) *Speed Characteristics*—An A.C. machine is a constant-speed machine, so that you are limited under all conditions to something within very close limits of full-load speed. With a D.C. motor on light load lifting or on long travel of the crane, it is possible to raise the speed to twice or three times normal full-load speed, with a great saving in time both in movement and in hoisting.

* The Proceedings at the Morning Session—*Discussion on Steelworks Locomotives: Diesel* versus *Steam*—were published on pages 87–106 of the *Journal*, January, 1947.

(4) *Control*—The possibility of dynamic braking with D.C. motors gives a lowering control which it is very difficult to obtain on A.C. drives, except by a good deal of complication. A further advantage is that the D.C. brake, in my opinion at any rate, is a much more satisfactory and reliable piece of apparatus than any A.C. brake.

(5) *Heating Rating*—Where you have frequent operations and reversals the D.C. machine is again pre-eminent, because the heating effect of rapid reversals is far less on D.C. machines than on A.C. machines, and for such duty it may be found necessary to put in a considerably larger A.C. machine than its corresponding counterpart on the D.C. side. The heating may be reduced by as much as 30% in D.C. machines as compared with A.C.

Reverting for a moment to the question of brakes, I think it will be found that in most cases D.C. brakes are fitted on A.C. cranes, which necessitates some form of rectifier unit.

(6) *Magnets*—Many steelworks cranes are fitted with magnets which with D.C. supply can be directly energized. With an A.C. crane, however, some converting unit must be added to give the necessary D.C. power.

(7) *Power Factor*—Most of our power agreements nowadays are on a kilovolt-ampere basis ; that is, we have to pay on the kilovolt-ampere rather than on the kilowatt consumption. With A.C. cranes we cannot expect to get an overall power factor greater than 0·65 to 0·7, with the result that expensive items of plant in the form of condensers or synchronous machinery must be installed for P.F. improvement.

(8) *Air-Gap*—The A.C. motor suffers from its small air-gap. This is not quite so serious today with roller or ball bearings, but it is a point which still has to be considered, as it compares unfavourably with the gap on the D.C. machine, which is considerably larger.

(9) *Single Phasing*—Single phasing may wreck the machine through overheating before the operator discovers that there is anything very much wrong.

(10) *Operator's Control*—With a D.C. motor a further movement of the controller or the cutting-in of additional contactors will result in a steady increase of torque. Such is not the case with an A.C. motor where too-rapid cutting-out of resistances will result in a stall, the torque reducing instead of increasing beyond a certain point. This demands a higher skill on the part of the operator.

(11) *Simplicity*—This claim for A.C. motors cannot now be upheld in practice, because in most instances it is necessary to use a slip-ring motor, which is slightly less complicated than the ordinary modern D.C. machine with its commutator. You have, in addition, to provide at least three T-bars as against two, and six cross-wires instead of four, and more are required where control refinements are added. Triple-pole contactors take the place of two-pole contactors. I shall come back to that later when dealing with maintenance.

(12) *Safety Factor*—In general, D.C. is less dangerous than A.C., and definitely so if you are using 230 V., which is preferred by some, as against 440 for A.C. Even with 460-V. D.C., as against 440-V. A.C., there is a greater safety factor from the personnel point of view.

Those are the main points in favour of D.C. motors, and I am left with three other matters to mention, all of which are very important : Maintenance, first cost, and efficiency.

(13) *Maintenance*—We have many hundreds of cranes working on D.C. and find that our maintenance costs are very reasonable. We have had motors running on heavy-duty cranes for ten years, which have never been off the crane for anything at all. Commutator trouble is almost unknown. If a machine does come into the shop after three years the commutator is sometimes given a skim-up, but otherwise it is left as it is. Brush wear depends on the conditions under which the crane is working, but we have motors in operation in which the brushes have not been replaced for periods of ten years. Maintenance costs with D.C. cannot be considered higher than with A.C., and in fact if anything we think that they are lower. One point is that if you do have "a bit of a splash" on a commutator, it is much more easily repaired than a similar splash on slip-rings.

(14) *First Cost*—With regard to first cost, the more I see of steelworks the more I am convinced that first cost should be to some extent a secondary consideration. When you realize the length of life that is got from electrical machinery, which after working for 30 years looks like running for another 30, a little difference in the first cost is negligible, and the best material should be chosen for the job.

We have taken out careful cost figures and based on a 460-V. D.C. installation as against a 440-V. 3-phase installation, including in the latter the necessary power factor improvement device and the extra T-bars and cross-wires, and comparing like with like—that is, the mill-type D.C. motor with the mill-type A.C. motor for the same duty—the actual cost of the D.C. installation is less than that of the A.C., even

taking into account the conversion from A.C. to D.C.

The use of D.C. at 230 V. did in the past involve a costly number of overhead wires, and cable costs were correspondingly high; but in the modern steelworks we put down rectifier substations at strategic points and bring the high-tension supply to them, so that our distribution losses are greatly reduced and cable costs are relatively small. Under these conditions, as I have previously said, the D.C. overall cost comes to a smaller total than the A.C.

(15) *Efficiency*—Though at first sight it may appear surprising, a close investigation will show that, if transmission and T-bar losses as well as conversion and machine efficiencies are taken into account, the efficiency of a D.C. installation is equal to or slightly better than that of an A.C. installation.

I have no grievance against the A.C. equipment for cranes. It will do all that you want it to do, but at the expense of simplicity. You can get something that approximates to dynamic braking on A.C. by the injection of D.C., by single-phase reversing, counter-current braking, or other means, but they all introduce complications. On an A.C. crane you can get D.C. mechanical braking by putting in a rectifier unit, and if you have a magnet crane you can deal with that by putting in a rectifier unit or a motor-generator set, but these are all expensive and rather cumbersome pieces of apparatus, which can be avoided by using D.C. You have to bear in mind that in most steelworks any big variable-speed machines will be D.C., and that if you are faced with the problem of converting a portion of your incoming power to D.C., there is not very much extra cost involved in increasing your conversion plant to deal with cranes. It is generally agreed that for live roll and screwdown drives D.C. is much to be preferred, and conversion for this purpose is consequently necessary.

Mr. J. Russell Taylor (Igranic Electric Co., Ltd.) (speaking on behalf of Mr. **R. A. West** of the same company): In examining this problem of A.C. *versus* D.C. drives for steelworks cranes, I have reached much the same conclusions as those which have just been given by Mr. Rothera. Speaking from the point of view of the manufacturer, there is no doubt that it is possible to design the A.C. drive to meet practically all requirements. I should like to restrict my remarks to the comparison between drives using a series motor on D.C., compared with a slip-ring motor on alternating current. There is very little which the A.C. motor cannot do almost as well as the D.C., but practically nothing which it can do better, except perhaps to regenerate. It would also probably be advisable to confine the comparison between the two motors still further, namely, to machines which are totally enclosed and 1-hr. rated. For evident reasons this specification is in vogue for the duties which we are considering.

Torque—In connection with torque I should like to give the following figures, which represent maximum values based on a commutation current limit for D.C. and a pull-out torque limit for A.C. These torques are slightly under the possible values which would correspond to 250% current for D.C. and 250% pull-out torque for A.C. with industrial motors, and to 350% current/torque for D.C. and A.C. mill motors:

	Industrial	Mill	
D.C. ...	3·0	4·5	Percentage of full-load torque.
A.C. ...	2·25	3·15	

The D.C. maximum torques are better and, as mentioned by Mr. Rothera, are obtainable independently of the maintenance of correct voltage.

Heating—In connection with the heating of the motor, I should like to add one or two points. As the overload increases with the D.C. drive, so the torque per ampere improves. Consequently the heating is less severe than would be the case if it were not for the advantage of the series characteristic of the motor. In this connection it is interesting to note that if two series motors of different power but equal rated speed are direct-coupled mechanically and series-coupled electrically so that their torques oppose, it is the smaller motor which will succeed in driving the larger motor backwards. It is assumed, of course, that current is applied and limited through a resistance in attempting the above experiment. With the slip-ring motor, however, the rotor current increases faster than the torque when the machine is overloaded. The stator current may not increase as fast as the overload (in torque) initially with some designs, notably the mill motor, but is always relatively greater in relation to full-load value than is the torque by the time the pull-out torque is attained. Consequently the A.C. motor heats up more rapidly than the D.C. under overload conditions, and it is advisable not to attempt to utilize the ultimate torque unless the duty cycle is very low. Figures 1, 2, and 3 show for 75-h.p., 750-r.p.m. synchronous motors designed for industrial, crane, and mill duty, respectively, plots of both stator and rotor current against torque expressed as a percentage. These figures clearly illustrate the above remarks.

One further point should here be emphasized: Many designs of 1-hr. rated, totally enclosed A.C. motors cannot carry even their magnetizing, or

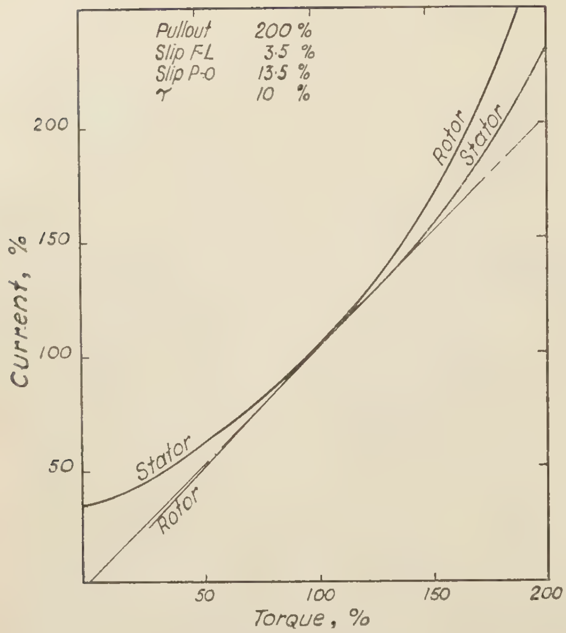


FIG. 1—Current/torque curves of industrial motor ; 60 min., 75 h.p. at 725 r.p.m.

no-load current continuously. This shows that periods of light-load running do not help the slip-ring motor to cool down, and demonstrates again that the series motor is to be preferred for cranes which operate on a frequent duty cycle.

Speed—With regard to speed, the control-gear manufacturer has always said, and correctly so, that it was much easier to give flexible control, especially on hoists, on D.C. rather than on A.C., but in the last few years various schemes have been produced whereby it is possible to obtain a control of hoisting and lowering speeds with A.C. which is more or less equivalent to that to which we are accustomed in the case of a D.C. dynamic lowering hoist. This is not the place to explain how this is achieved, but, generally speaking, as Mr. Rothera says, the apparatus is rather more complicated.

It is well known that the A.C. motor cannot run beyond its synchronous speed unless it is driven round by the mechanical load. This condition, which is referred to as an overhauling load, exists in practice only when a fair load is being lowered on a crane hoist and that is not an occasion when a high speed can be considered a real advantage.

With cranes, the most time is used or saved during the operation of the long-travel motion for overheads, the cross traverse for ore bridges, and of course the hoist for both types. All these motions tend, if properly designed for a duty cycle, to spend much of their time with the motor lightly loaded, when the series motor will show to great advantage.

With particular reference to the long-travel drive of overhead cranes, I should now like to give some figures which will emphasize the advantage of the series motor compared with the slip-ring. They are embodied in Table I, which has been extracted from distance/time performance curves.

The crane structure weighs 80 tons and all drives are designed for the same rate of acceleration and braking. The times which are tabulated for travelling the various distances are those required if the crane starts from rest at one point and until it is brought to rest at the stated distance further down the shop.

The significant fact emerges that a 70-h.p., D.C. drive geared for 300 ft./min. has the same performance for all distances up to 60 ft. as a 140-h.p., A.C. drive geared for 400 ft./min. Beyond 60 ft. the D.C. drive has a progressively better performance. Furthermore a 100-h.p., D.C. drive geared for 400 ft./min. has a much better performance than the A.C. for all distances beyond, say, 50 ft. The horse-power for these drives has been obtained with due regard both to heating and to torque and keeping to our original specification of 1-hr. rating and total enclosure.

Power Consumption—As makers of control gear we occasionally are taken to task on the

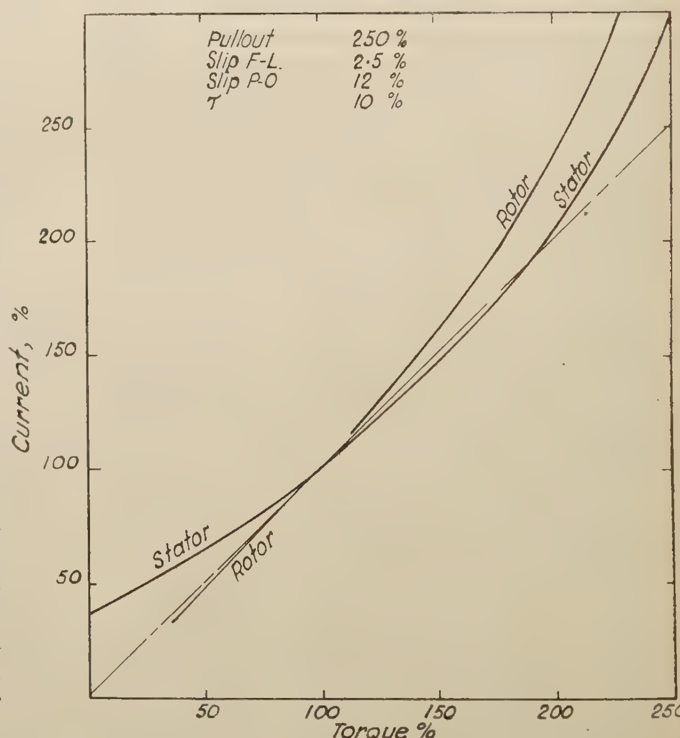


FIG. 2—Current/torque curves of crane motor ; 60 min., 75 h.p., at 730 r.p.m.

TABLE I—*Comparison of Slip-Ring Motor with Series Motor*

Type of Drive	Horse-power	Geared Speed, r.p.m.	Time, sec., to travel: 10 ft.	20 ft.	40 ft.	80 ft.	200 ft.	800 ft.
A.C. ...	140	400	4·2	5·9	8·9	14·9	33	123
D.C. ...	70	300	4·3	6·0	9·0	14·4	30	98
D.C. ...	100	400	4·2	5·8	8·4	12·8	26	77

question of the power consumed with various schemes which provide slow-speed lowering. I think it is fair to state that it is not possible to get creeping speeds without wasting power, for, even if you have recourse to a Ward-Leonard set, you have to keep that set going during considerable periods when it is not actually in use. This fact comes to our rescue somewhat in connection with the wastage of power with what might be termed 'control-gear schemes.' In practice, crane hoists are not operated at slow speed except momentarily for picking up and putting down

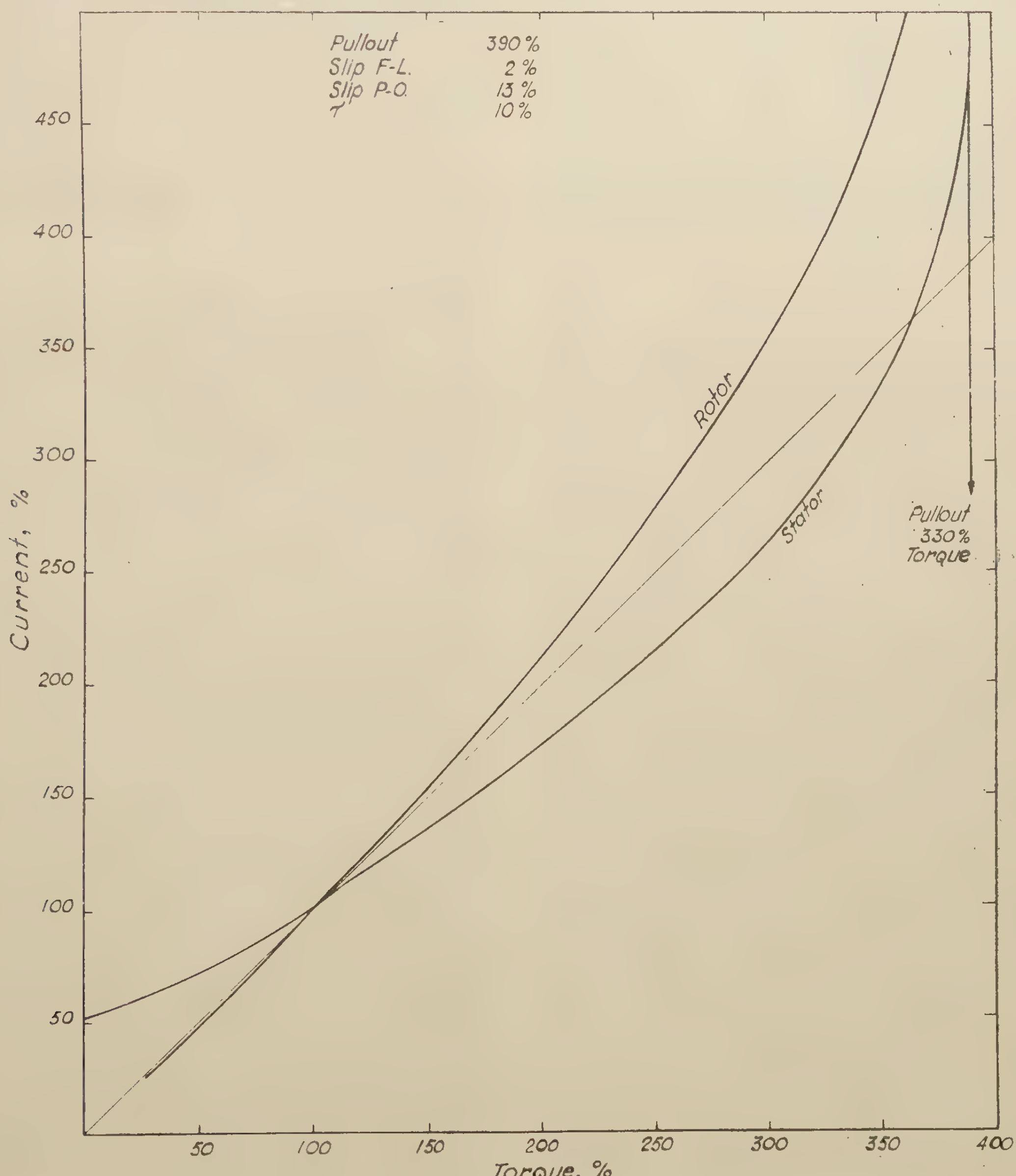


FIG. 3—Current/torque curves of mill motor: 60 min., 75 h.p. at 735 r.p.m.

loads. There are exceptions, as in foundries, but in the majority of cases the creeping speeds are only in use for a very short time, so that the relatively high consumption of power is not just as important as at first sight might seem to be the case.

However, I thought it might be of interest to show a rough comparison of the power consumption of A.C. and D.C. 'control-gear schemes' and for this purpose I have chosen the case of a dynamic lowering hoist *versus* an A.C. job controlled by counter-current lowering. Table II shows that there is little difference between A.C. and D.C., except that when lowering at full speed and fully loaded, the A.C. job is able to regenerate a great deal more power than the D.C.

TABLE II—*Power Consumed when Lowering*

	20% Speed	50% Speed	Full Speed
A.C., light hook ...	40%	20%	10%
D.C., light hook ...	95%	70%	70%
A.C., fully loaded ...	110%	90%	−70%*
D.C., fully loaded ...	110%	75%	−15%*

* Regenerating

Dynamic Braking—With regard to dynamic braking, it is usually emphasized that D.C. has the important advantage that this form of electrical braking is always available, even when the control gear is in the 'off' position. Personally I am inclined to think that this is an overrated advantage. It is undoubtedly an inherent safety feature whose benefit cannot be denied, but I doubt whether it is often called into action.

It is often not realized that a D.C. hoist does not give dynamic braking in the hoisting direction. Owing to the high speed of the lightly loaded motor, there is much stored energy in the rotating parts when the hoist limit switch trips. As there is no dynamic braking in this direction, all of this energy must be dissipated in the brake. The dynamic braking series limit switch can be used instead of a shunt limit switch ; the former changes over the connections so that dynamic braking is available in the hoisting direction. This type of limit switch has saved much money in avoiding expensive breakdowns caused by running the pulley block into the winding drum. The dynamic brake limit switch enables more headroom to be obtained, as it is possible, with safety, to set this type of limit switch a great deal closer to the position of maximum lift than is the case with a shunt limit switch. It provides an almost certain guarantee against a serious overwind, and with D.C. mill motors the surge of current when the motor, acting as a generator, passes its initial peak current is quite harmless. With industrial motors there may be a splash on the commutator or even a catherine wheel, but these are much more easily put right than serious damage to the machinery.

With the A.C. hoist, of course, one cannot use a dynamic braking series limit switch, but then the need for it is much less. Roughly speaking, the stored energy of the A.C. job is only about 30% of that of the D.C. when operating at maximum hoist speed.

The principal original advantage of dynamic braking after lowering was in the enormous reduction of wear and tear on the brake linings. All first class A.C. installations now embody a system of electrical braking whereby the load and connected mechanism are retarded principally by the motor, and the electro-mechanical brake is applied only after the speed has been reduced to under 25%.

Turning now to the comparison of A.C. and D.C. as applied to drum controllers, magnetic contactor equipments, and brakes, there is still a preference for D.C., particularly when required for heavy duty.

Drum Controllers—Dealing first of all with the drum controllers, the arcing in A.C. drums is less than in D.C., and it is consequently possible to use this type of control up to a higher horse-power on A.C. The great number of contacts which are required for three-phase operation increases the size of the A.C. drums ; this is particularly noticeable if (*a*) the rotor resistance is cut out in balanced steps and/or (*b*) the rotor current is a great deal higher than the stator current. In fact, with increasing power it soon becomes impossible to retain balanced rotor control and it is usual to cut out the resistance in unbalanced steps in order to avoid an unwieldy size of drum. This unbalanced rotor control increases the relative heating of the motor and must be avoided on duty-cycle applications.

On D.C., the destructive effect of the arcing in drum controllers makes their maintenance a difficult problem at relatively small horse-powers. Consequently the advantage of magnetic control is brought into play at relatively low power. With A.C., on the other hand, drum controllers may be used up to a higher power except when the duty makes it advisable to use balanced rotor control and also introduces a need to prevent the resistances from being removed from the circuit too quickly. If the drum control is advanced through its notches faster than the motor is able to accelerate, as Mr. Rothera has pointed out, the current (both stator and rotor) increases without proportionately increasing the torque ;

in fact it is quite easy to go so far that the torque is actually reduced whilst the current is still increasing. This is a very prevalent cause of overheated A.C. motors and can best be prevented by the use of magnetic control wherein the rate of closure of the contactors is automatically held back by current-limit or preferably by time-limit supervisory apparatus which is readily and almost universally fitted to the contactor gear.

Contactors—Modern A.C.-operated contactors of good design are more hardy than their forebears, but they still have inherent defects when compared with D.C.-operated contactors. The A.C. coil can burn out if any mechanical obstruction prevents the contactor from getting right home. Furthermore, if the voltage is low the contactor may fail to close properly and this condition will also burn out the coil. Dirt can accumulate on the magnet faces and cause severe noise and chattering. Shading rings may come adrift or be broken with similar results. Chattering reduces the life of the magnet if maintenance is not properly carried out. Because an alternating magnetic flux in a piece of plain iron or steel produces intense heat on account of the induced eddy currents, it is necessary to use a laminated construction for the magnets of A.C.-operated contactors. The laminations are riveted together under considerable pressure but, even so, the construction is inherently less robust than the simple one-piece castings which are used with D.C. Consequently we recommend D.C.-operated contactor gear for heavy-duty A.C. cranes, with the attendant advantage of inductive-time-limit control. This ITL system, as it is called, gives sequence and timing of contactors without relays ; as the control is obtained from the time-constant of the contactor itself, it is both permanent and simple.

Regarding the maintenance of copper tips on A.C. and D.C., I think it is undoubtedly true that for manual controllers of equal power the D.C. consumes a great deal more copper than the A.C. However, with contactors, we are under the impression that there is nowadays but little to choose. We have increased the efficiency of operation of the D.C. blow-outs in two stages over the last quarter of a century and at each stage the life of the D.C. copper was increased by some 200–300%. Consequently we are uncertain whether it can still fairly be claimed that A.C. copper maintenance is less than D.C. It is one of those questions where results in the field are very hard to obtain on a strictly comparative basis of equal duty, equal power, and equally modern apparatus.

Brakes—The brake is a very important part of a crane hoist and if the type of electrical operator takes the form of a magnet, D.C. is superior to A.C. to a very marked degree. All those comparative features, which have just been described in connection with the magnets for contactors, apply equally to the magnets for brakes, but with more emphasis on the defects encountered through laminated construction, shading rings, failure to close, and so on. The higher the pressure of riveting used for the construction of the laminated A.C. magnet, the better it works, but, on the other hand, the greater is the danger of the rivets breaking due to fatigue. A brake is one of the few mechanisms in which a very great force moving through a very small distance is the basic characteristic. This happens to be a characteristic which is also inherent in the D.C. magnet. An A.C. magnet cannot be made to give an efficient output if it is asked to operate only through a short stroke. Consequently the best designs of D.C.-operated brakes comprise a magnet system in which the armature moves only a very small distance. This produces a brake which is quiet in action and the absence of hammer-blow naturally results in long and trouble-free life. The reliability of the brake is also improved by the absence of a system of levers and cranks which is required to obtain a great force through a small distance from the A.C. magnet.

The adoption of an A.C. torque motor for brake actuation was one successful but expensive solution. Best practice nowadays seems to be divided about equally between the use of D.C. short-stroke brakes with rectifiers and thrustor-operated brakes. Both of these types are successful, but the thrustor, like the A.C. magnet, is essentially a long-stroke mechanism.

The relative angular movement of the parts of a D.C. brake can, by good design, be kept to within a few degrees, and the mechanical maintenance of pivots ceases to be any problem at all.

Another point in favour of the D.C. brake is that crane machinery can be designed for the installation of the same brake whether the supply is D.C. or A.C. ; when used with a D.C. motor the brake can be series wound, which is a major advantage, as it can then be guaranteed that there is torque on the motor available to sustain the load before or at the same time as the brake is released.

In conclusion I should like to leave with you the following thought : Steel mill cranes and ore bridges inevitably include a relatively high proportion of duty-cycle jobs. For these, D.C.-operated contactors and brakes are preferable. For minimum maintenance, reduction of spares and simplicity, a good case can be made

out for utilizing D.C. contactors and brakes throughout an A.C. mill. Such a policy involves an increased first cost, but will result in a more contented "electric shop" and the additional cost will actually earn a high dividend in the form of reduced cost of maintenance and renewal.

The **Chairman** : Mr. Parker will put forward an alternative view, particularly with regard to large ore bridges.

Mr. **J. Parker** (Messrs. McLellan & Partners) : Some day I must ask Mr. Russell Taylor to assure me that an A.C. hoist with special control is as good as a D.C. hoist. I feel that it is not, but perhaps he will have more to say about it. He pointed out that the D.C. contactor is more reliable than the A.C. mechanism. D.C. contactors can be used on A.C. drives by employing a small motor-generator set or a dry-type rectifier to give D.C. power for control circuits. I have found both methods reliable and satisfactory.

I should like Mr. Rothera to explain in more detail why 460-V., D.C., is not as dangerous as 440-V., A.C. In any case, when on a crane, contact with live wires usually leads to a fall, and it really does not matter what the voltage is.

In his comparison of costs, Mr. Rothera referred to power-factor correction for A.C. cranes, and he included the cost of static condensers in making his comparison. Surely, power-factor-correction in a steelworks must be considered, not for cranes alone, but for the whole plant, and it is usual to employ large synchronous machines for this purpose. Therefore, to include power-factor-correction plant in a comparison of costs is, I think, a little unfair to the A.C. crane.

We have had very satisfactory experience with dry-type rectifiers feeding magnets on A.C. cranes.

I think that at last almost everybody is beginning to realize that the electric brake is not a service brake, and is useless for controlled braking. It is only a holding brake. The need for controlled braking on travel motions is now being met by the electro-hydraulic brake in this country. The plain hydraulic brake has been in use on American cranes for a number of years.

With regard to the first cost of a crane, the cost of the electrical gear is only 15 to 35% of the total cost, and it is essential to instal the most reliable electrical apparatus available. When, for example, a locomotive breaks down another can be brought in to do the job, but this is not possible when a crane fails. I agree with Mr. Rothera, therefore, that first cost of crane electrics should not be a prime consideration.

To refer to power supply, the ease with which A.C. can be transmitted makes it most desirable to employ it wherever possible. The introduction of the rectifier has reduced A.C.–D.C. conversion losses, but even so, A.C. is better for distribution. Large D.C. cranes and ore transporters introduce difficult switching and transmission problems. I recollect seeing some ore bridges in America operating on 200-V., D.C. (their standard D.C. voltage), where 16 sq. in. of copper were used to give the supply to the collector rails. I wondered whether they were correct in choosing D.C. for this application.

There is no doubt that the series motor gives a superior performance to the slip-ring machine, but the choice depends on the duty of the crane. A.C. motors work well on slow-moving cranes, but it would be difficult to employ an A.C. hoist motor on a ladle crane, for example. However, I suggest we might mix A.C. and D.C. on such cranes, so that A.C. transmission can be employed, A.C. motors used on the travel motions, and Ward-Leonard control applied to the hoists.

There is no doubt that Ward-Leonard control gives faster acceleration and smoother control, compared with any other method. On a ladle crane, the motor-generator set could be accommodated on the bridge and used to feed the main hoist and the auxiliary hoists. The travel motions could be equipped with slip-ring motors. The supply brought to the crane gantry might be at high voltage—3,300 V. might be employed, with a transformer on the bridge for the travel motions and lighting and heating circuits. In other words, use D.C. where it is necessary and A.C. where it is applicable, transmitting the power required at a high A.C. voltage.

This also applies, perhaps even more strongly, to ore bridges. The man trolley has to work as fast as possible, because we are interested in the turning round of ships, but the bridge has only to move occasionally.

I suggest the man trolley is an ideal application for Ward-Leonard control, using it on the holding line, closing line, and cross traverse. The travel motors, emergency brakes, air compressor, and the boom hoist could be equipped with A.C. motors, and the lighting could be on A.C. The supply could be brought to the bridge at 3,300 V., the generator-set motor operated at this pressure, and a transformer installed on the bridge to give a low-tension A.C. supply for the lighting and other motions.

Mr. **G. Wright** (Messrs. Ashmore, Benson, Pease, & Co., Ltd.) : In my opinion the characteristics of D.C. drives are required for high-capacity ore bridges and transporters.

To instal Ward-Leonard control not only adds to the cost of the electrical equipment, but there

is also an additional cost for steelwork owing to the extra weights that have to be carried. The question of power supply available must also be taken into consideration when assessing whether the additional cost of Ward-Leonard control is justified.

Mr. **W. Redfern** (The Lancashire Steel Corporation, Ltd.): All the speakers on this subject have told us of the advantages of D.C. and the disadvantages of A.C., so that I cannot see that much further argument is necessary. I am surprised that we are trying to forsake the old series-wound D.C. motor. There is no more accommodating a machine for torque and speed, that has all the advantages and none of the disadvantages of A.C. I have had experience with both kinds for about 30 years, and am still in favour of the D.C. series-wound motor.

Mr. **F. Somers** (Messrs. Walter Somers, Ltd.): I should like to speak from the point of view of the user.

We have had 40 years' experience on D.C., and only 30 with A.C. because A.C. was only recommended to us then.

I am speaking for the forge departments of a steelworks where electric overhead cranes are used, which have heavy duties to perform. We have 16 cranes working on D.C. and 11 on A.C. In the electric overhead cranes we look for output and do not consider the initial cost; the cranes have to work day and night and keep on working. The result is that after our experience we are absolutely in favour of D.C., and I have taken the trouble to travel 100 miles to speak in favour of D.C.

I maintain that with properly designed cranes and crabs and with covered-in gears with modern lubrication, for heavy duty combined with accurate driving, there is no comparison between D.C. and A.C.

I prefer D.C. motors of the mill type which are specially designed for crane work. For longitudinal travel, I would put forward controllers designed for plain series with power and rheostatic braking, and for the hoist motors, plain series motors with slow speeds on the first two notches with potentiometer control and dynamic braking for lowering. At one moment the crane may be hoisting a load of 60 or 80 tons, and the next moment the hook may be travelling light.

Mr. **J. Baker** (Messrs. Joseph Booth & Bros., Ltd.): It is time that the crane maker had something to say. There is no hard and fast rule to decide between A.C. or D.C.; it depends on circumstances. I feel, with all due respect to you steelmakers, that you are taking a very narrow view, because you have really been discussing your cranes which we call heavy steelworks cranes, which are roughly speaking, process cranes perhaps from 5 tons up to 100- or 150-ton ladle cranes. As far as those cranes are concerned, there is no doubt that D.C. is the best proposition, and I agree with Mr. Somers.

I do not altogether agree that you cannot get the control you want on A.C.; I think that you can; but D.C. is the better proposition, because it is simpler, and, comparing like with like, and bearing in mind that on an A.C. crane you must have bigger motors to give the same equivalent speeds as with D.C., it is cheaper to use D.C. I am not convinced that it is cheaper in maintenance; I think that is a moot point, and I have to be swayed by the views of users.

There is, however, a case for A.C. cranes. There are many smaller cranes about a steelworks which are not process cranes. For these, the A.C. crane is much cheaper in first cost and in maintenance cost. We have to consider these costs. It is not everybody in the steel industry who will be nationalized and have unlimited money to spend on plant; we have to consider the man who is running the works on his own capital.

Again, when we get to very big cranes, such as ore bridges of, roughly speaking, above 100 h.p., wiring becomes a huge problem on D.C. When you have a 300-h.p. motor running off 230-V., D.C., the wiring problems are not insurmountable, but they are getting on that way, and then A.C. comes back into its own. Again, on an ore bridge the cycle is known in advance; you know what you want it to do. It is the same cycle over and over again, and you can plan your speeds to suit that cycle. It is the time taken to do the job that counts, not how fast the crane goes with a light hook and a heavy hook, and it is possible to get A.C. cranes to do the job in the same time as D.C., so that on the very large cranes we again have a case for A.C.

I have always felt that a ladle crane would be a case for A.C. if it was the only crane in the building, but it never is. You use D.C. because it suits your lighter process cranes. A ladle crane is slow moving, and is a case where A.C. control could be put in to give absolutely the same features as D.C. One of the problems on A.C. cranes is the extra number of cross-traverse collectors, especially with an auxiliary crab. On a D.C. crane you have 22 collectors with two crabs, but on an A.C. crane you have something like 28, and getting them in when you use T-bars is a problem.

No mention has so far been made of the A.C. commutator motor. I am not advocating it, but it does give the nicest form of variable speed

control that there is. To get the necessary torque at slow speeds necessitates a motor of very large overall dimensions ; housing this motor is a problem.

In three or four years' time we may look back on this meeting and wonder why we ever discussed this subject at all, because electronic control may have been developed by that time, and we may be able to bring A.C. on our crane and convert to D.C. on our rectifier and get all the control we want.

Dr. **L. N. Bramley** (The British Iron and Steel Research Association) : I was interested in Mr. Parker's remarks about the electromagnetic brake not being the brake to use on the long-travel crane. He advocated the use of the hydraulic brake. The use of a hydraulic brake is not, in my opinion, a complete answer, because it cannot be used in all the applications in which the brake is required. You may split up the travel-motion brakes broadly into two classes, those in which the point of application of the brake and the point at which it is actuated suffer relative motion and those in which they do not. For example, an ordinary electric, overhead travelling crane with a fixed cab has no relative motion between the position of the foot pedal and the long-travel brake, but the crane which has the cabin moving has relative motion between these points, and the hydraulic brake is no longer applicable.

It seems to me that an electric brake is needed which will have universal application to both types of crane. Neither the D.C. electromagnetic brake nor the A.C. thrustor-type brake is applicable, since they are both spring-actuated, and thus snatch when they go on, so that inching is not easily achieved.

There is a field of application here for the torque motor brake. These brakes have been made for other purposes than for cranes, but they seem to have been dropped. I should like to know whether there is any particular reason why a torque motor brake, which has a constant-length air-gap on it, should not be used in this application.

The recent developments in A.C. commutator motors have enlarged their field. I do not know whether there is any possibility of applying them to cranes as well.

A recent development in America has been to control the speed of A.C. motors by putting a series resistance in one phase. I should very much like some information on the range of speed control which can be obtained by such means.

Mr. **Russell Taylor** : I should like to answer the last point raised by Dr. Bramley. On A.C. crane hoists it is now possible to obtain speed control on the lowering side and also on the hoisting side without the use of a mechanical load brake. There are various ways of doing it. The method with which I am conversant for getting some sort of speed control on the lowering side is by reversing the motor. In other words, you allow the load to pull the motor round faster and faster when the motor is connected in the hoisting direction. By suitable adjustment of the rotor resistances, it is possible to obtain speeds below synchronism. In other words, you adjust the rotor resistance to give a hoisting torque which balances the load torque. In such a case with anything from about one-third load to full load you can obtain slow lowering speeds without the use of the load brake, but by means of juggling with the controller.

From that, another scheme was produced whereby the juggling with the controller was taken out of the operator's hands and taken over by a series of relays responsive to the motor volts. Crane hoists have been built using that scheme and are now on the market, and by that means it is possible to obtain five stable lowering speeds, the bottom speed being 10 to 15% of normal full-load hoisting speed. It is possible to obtain a creeping speed on the hoisting side with any load from no load to about one-half to three-quarters load, depending on the efficiency of the gearing. Those methods were produced in Great Britain.

While those methods were being developed here, the Americans thought of the possibility of unbalancing the stator of an induction motor, either by inserting a resistance in one of the three stator leads or by varying the voltage directly by means of, say, an auto-transformer. If the stator is unbalanced the motor behaves like two motors, one trying to go forward and the other backwards ; there is a tussle between the two, and you get a series of straight-law curves by varying the unbalance, more or less the same as on the lowering side of a dynamic-lowering crane hoist. Unfortunately the slope of these speed torque curves is rather greater than in the D.C. case, unless you use a definitely oversized motor, but it is quite possible to use the system, and it has been used in the U.S.A. ; in fact, I know of some of these controllers being used on ladle cranes. If I invented something new, I would consider a ladle crane the last place to try it out !

I should like to have more information concerning Mr. Parker's suggestion of putting a Ward-Leonard system in for control of the duty-cycle drives on an ore bridge. This system would give very good control but it would be necessary to have three generators so that individual control is

obtainable for holding line, closing line, and cross-traverse motors; presumably these generators would be driven by a single A.C. motor. I think that he would have great difficulty in making room for these machines on the man trolley, where they really ought to be located; also they would add weight to a part of the design where it is desirable to keep weight to the minimum, in order to avoid increasing the traverse power. The increase in live load would add to the weight of the bridge. If, alternatively, the machinery is placed at one end of the bridge, you would require a pair of main leads from each generator, and shunt leads for fields, brakes, etc., and I have heard rumours from steelworks that shunt leads are an abomination, especially if they have to be taken through T-bars; they have a habit of failing to make contact through dirt.

I should also like to join issue with Mr. Baker when he says that for an ore bridge, A.C., if the motors are the right size, would be just as good as D.C. Unfortunately, on an ore bridge it is difficult to stop the trolley. You want to run up to the end as fast as you can and stop in the shortest possible distance. With A.C., if there is a failure of power you will lose control and have to rely entirely on your brakes, which may not be in good adjustment. With D.C., on the other hand, it is possible to get automatic braking.

I know of a very early ore bridge controlled on A.C. The cross-traverse was by two large A.C. motors, and the speed was about 400 ft./min.—fairly high—and very large buffers were put on to stop the jar if you happened to overrun the limits. One of the provisos was that it was to be possible to run into these buffers at half speed. I was the unfortunate person who had to drive the crane, and the consulting engineer was sitting behind me. I started off gently and went faster and faster until I said that it was half speed. He said it was not. I told him I would drive it at half speed if he would remain on the crane, but we both went down. On D.C. that would be entirely automatic; on A.C. it was supposed to be but was not.

The **Chairman**: I think that Mr. Parker cut his remarks to the very bone, and he should have an opportunity of explaining what he has in mind in greater detail. I think that it will then be seen that there is more in his views than there may appear at first to be.

Mr. J. Parker: Perhaps I might describe an installation on which we are working at present. A wharf is to be equipped with three 12½-ton ore bridges, with a possible fourth in the future. Each bridge will have a 200-h.p. motor on the closing line, a 200-h.p. motor on the holding line, and two 150-h.p. motors on the cross-traverse, giving a total installed horse-power of 700 on the man trolley. All these motions will operate together, and the peak load will be about 1,200 h.p. Under normal conditions the three bridges will be working at the same time.

If a 440-V., D.C. supply is used, the collector rails would be formed from 130-lb. rails, reinforced with 4 sq. in. of copper, and the feeder cables would have a total section of 5 sq. in. per pole. The supply would be obtained from two 750-kW. motor-generator sets with one stand-by set located in a building at one end of the wharf. The total cost of the collector rails and supply is roughly £40,000.

If we consider an A.C. supply, we must use D.C. on the holding and closing lines and on the traverse motions (in spite of the remarks of Mr. Russell Taylor) to obtain high operating speeds. We will make these motions Ward-Leonard. The motor-generator set would not be located on the bridge because of difficulties with the control circuit collectors, but incorporated in the man trolley. This increases the gross weight of the man trolley by about 15%.

Is it worth while to carry this extra weight? We submit that the advantages of Ward-Leonard control over the standard D.C. will compensate for this; the man trolley can operate faster for the same stresses in the bridge, losses of stationary converting plant are avoided, and mechanical and electrical maintenance of the bridge will be considerably reduced.

The A.C. supply is quite simple. Power would be brought to the wharf at 3,300 V. In this case there is a substation near each end of the wharf, enabling a duplicate supply to be brought to the collector rails, and the rails can be sectioned.

The copper sections are much smaller; a 3 × ⅜-in. angle-iron will be used for each rail and each feeder cable is one 3-core, 0·25-sq. in., and the total cost is about £11,000.

Thus, on supply alone, there is a saving of about £30,000 which will more than cover the additional cost of Ward-Leonard control on the three bridges.

To refer again to the extra weight of the man trolley, ore bridges have been built for very much larger capacities than the case in question, so we can assume there are no structural difficulties involved in our application. The use of high-tensile steel for some of the structural members, light-alloy sheets for the man-trolley enclosure, "submarine" construction on the motor-generator set, and so on, all tend to offset the effects of the additional weight of electrical equipment which has to be carried.

I shall be interested to hear the views of other speakers on these suggestions.

Mr. **P. F. Grove** (Messrs. John Miles & Partners (London), Ltd.) : I feel that I am not a true steelworks engineer, having spent half my time on the grid, but being responsible for the electrification of two large integrated iron and steel works in this country, I have discovered that there is more in this A.C.–D.C. problem than I thought at first.

Of the two steelworks, the first was intended to follow the German tendency of about 1933 of saving conversion losses by adopting A.C. throughout, in which respect they were well advanced in A.C. types of motor. I arrived after the first section of the plant was running, and there was not a great deal of trouble, as far as cranes were concerned. The stators are inclined to slip in their casings, though I do not know why the makers do not weld a bar of steel across the back to stop it. A.C. contactors require more maintenance than D.C., and this takes me back to the power side of my work.

From 1928 onwards, the national grid system has been spreading around the country, and that very insidious and unpleasant short-circuit characteristic of A.C. has come nearer to the machinery in many of our works. This makes it necessary to examine fundamentally the difference between A.C. and D.C., and owing to the frequency there is no doubt that heating with A.C. is greater than with D.C. I had some experience with heating of heavy rolling-mill auxiliaries, which are similar to crane drives, and although A.C. motors can be made to do the same duties as D.C. motors, I agree with Mr. Rothera that you cannot get the same overload capacity for heating.

Mr. Rothera also raised the question of safety and, although A.C. is more dangerous than D.C., I should not like to say that 460-V., D.C., is not any safer than 440-V., A.C., if you are standing on a ladder and come in contact with it, as a previous speaker has said. Again, I think it is a question of the fierce short-circuit capacity behind the A.C. system that is important, whereas D.C. has to come through some means of conversion and in that process it is what I call ‘ softened.’

I do not want to side violently with D.C. because I think it is a question of transition. In a works which is nearly all A.C., the conversion losses are perhaps less, but you have to look at the matter from a broad point of view and if you have to lay out a new steelworks on a green field, it is not just the cranes that have to be considered ; there are many other drives. I have tried to solve this problem in my own mind by dividing the drives into constant-speed and variable-speed and looking at it from the point of view of distribution, which Mr. Parker touched on. Constant-speed drives should be A.C., because that is the simplest and cheapest way of conveying power to them, but when it comes to variable speed, I think that you have to treat every case on its merits. For instance, on a blast-furnace hoist, Ward-Leonard control for a heavy duty-cycle drive is undoubtedly the best that we have found so far.

When you come to cranes they depend to some extent on the other drives in that part of the works. If you have a shop some distance out from the rolling mills and a crane not doing heavy duty there is no object in putting in conversion for it, but in the steelmaking plant and rolling mills the A.C.–D.C. question enters in with local auxiliaries. It is my opinion that heavy auxiliaries round a mill should definitely be D.C., preferably with Ward-Leonard control, which is A.C. from its source of supply, but there are other auxiliaries not quite so important where I think that D.C. has an advantage. In that case, as you have to convert locally, you may as well convert for the cranes and treat the whole section as a variable-speed unit.

Turning from this general aspect back to the second steelworks, where D.C. was available from the start, I have found that the maintenance and simplicity of control in the melting shops and soaking pits presents a much easier problem, in which case I would side definitely at present with D.C. for heavy work. Where you have lighter duty outside the heavy shops, A.C. is convenient and economical for distribution.

In connection with Ward-Leonard drive for cranes, I am surprised that Mr. Parker did not mention those on the Fontana Dam construction.

I do not know why you all laughed when commutator motors were mentioned. It depends on the type, and although you may not like the Schrage commutator motor with brush-shifting gear for cranes, there is another type with an induction regulator, which is being developed for heavy steelworks duty. You may find that the true variable-speed A.C. drive is in embryo, and in a few years' time may go far towards solving this problem.

Mr. **W. J. Pool** (British Thomson-Houston, Ltd.) : Mention has been made of the use of A.C. commutator motors on cranes. We have applied a considerable number of motors of the Schrage type over a number of years to fitting out and dockside cranes, and they are operating very satisfactorily on these duties. Their characteristics are as good as those of a Ward-Leonard system. I am not, however, advocating their use for steelworks production cranes for the reason

that it is not practicable to build them with the extremely rugged mechanical features associated with the D.C. mill-type motor ; yet they may well find a place in the steelworks machine shop or roll-turning shop.

Mr. Russell Taylor mentioned the unbalanced phase scheme for obtaining characteristics in an induction motor comparable with those obtained on a series motor under diverter control and explained that it is equivalent to having two motors acting in opposition. Unfortunately a motor used in this way also takes as much current as two motors, and the resultant heating is likely to rule out the scheme for cranes engaged on continuous production work.

Mr. Grove remarked that A.C. mill auxiliary motors heat up more than D.C. motors on similar duties. I think that this is partly due to the illogical method of rating used in this country and also in the U.S.A. It is usual to specify such motors by a short-time rating, *e.g.*, 1 hr., which means that the losses generated in the machine must not heat it up beyond a certain specified temperature rise when it is run at nameplate rating for 1 hr. The chief thing needed to meet this requirement is that the machine shall contain a sufficient mass of metal to absorb the heat generated. Now, the normal steel mill auxiliary duty is not like this at all, but consists of a continuous cycle of loading and the quality required of a motor to meet this is the ability to dissipate heat rather than to store it. It is more difficult to get heat away continuously from the A.C. mill-type motor than from its D.C. counterpart, if both are designed on a short-time rating basis, owing to the fact that the former machine is more full of active material and has, in consequence, a much poorer internal air circulation. Another factor which is important on peak loads is that the current increases more rapidly at increased torque in the case of an A.C. motor than it does in the case of a D.C. series motor.

Coming now to the question of Ward-Leonard control, I should like to endorse the Chairman's comment that as this system is standard practice for large excavators it deserves serious consideration for ore bridges. The space problem arises on excavators also and it has been met by the design of motor-generator sets of specially shortened construction having a minimum number of bearings. They are also designed to withstand the same kind of mechanical shocks as those to which they might be subjected on ore bridges. A further feature used on excavators, which might be applied with advantage to cranes and ore bridges, is the generator with inherently self-protecting features, rendering current-limiting relays unnecessary.

On the general question of A.C. *versus* D.C., I should like to stress a point that was implicit in some of Mr. Rothera's and Mr. Parker's remarks. It is rarely true nowadays that A.C. and D.C. are equally available. Power is almost invariably generated or purchased as A.C., and if a constant-voltage D.C. system is installed, then the capital, running costs, and the maintenance of the converting plant should be debited against the D.C. motors. I do not suggest that this is enough to tip the balance in favour of A.C., but it should not be lost sight of. This view of the matter, coupled with the fact that A.C. is more convenient to distribute, tends to support Mr. Parker's suggestion of using A.C. for the duties for which it is suitable and Ward-Leonard for the others. There will still be a considerable field for constant-voltage D.C. which should be obtained from local converting stations, as suggested by Mr. Grove, rather than distributed from a single central substation.

Mr. **J. L. Gaskell** (Appleby-Frodingham Steel Co., Ltd.) : As far as heavy steelworks cranes are concerned, I am quite satisfied that we are doing the right thing by continuing with D.C., particularly for melting-shop cranes, hot-metal cranes, and the fast mill cranes. With regard to A.C. contactors, Mr. Russell Taylor asked for practical experience of contact life. I have not had any accurate lives taken, but my impression is that there is no marked difference in contact life between A.C. and D.C. Considering the iron circuit, however, there is a marked difference. We have D.C. crane contactors in use which are perfectly sound after twenty-five years' use, but the A.C. iron circuits have had to be renewed after about three years, working on twenty-one shifts a week.

It has been said that the shading ring stops the chatter of A.C. contactors. That is rather an optimistic view for crane contactors. The biggest difficulty with the laminated iron circuit is that of the constant hammering of contactors on reversing duty ; the laminations splay out and eventually have to be replaced. If I were repeating those particular jobs, namely, grab-cranes, I should have the contactors D.C. operated.

There is another point in connection with D.C. at 220 V. (the system on which we operate) which has not been mentioned. It comes within the scope of Exemption I of the Electricity Regulations, but I would suggest that we should not lean too heavily on that for crane work, because there are other factors with regard to safety on cranes which rather override Exemption I.

On the general question of A.C. *versus* D.C., we have, as has already been mentioned, to consider very carefully what plant we put in for

conversion, particularly in these days of high fuel costs and low fuel availability. It is my experience with rotary machines, La Cour converters, and synchronous motor-generator sets, that our all-week efficiencies are such as to give us something of the order of 33% losses—expressed as a percentage of the D.C. output—which means that a considerable amount of power is going to waste. I am making use of the opportunity which now occurs by putting in steel tank pumpless rectifiers, and I am expecting to cut these losses considerably. At the same time that we cut those power losses, we shall also cut the maintenance costs very considerably.

I have not given careful consideration to the application of the Ward-Leonard system to ore bridges. Our ore bridge is operated on 220-V. D.C., with one 250-h.p. motor on the holding line, one on the closing line, two 80-h.p. motors on the cross-traverse and four 40-h.p. motors on the long travel; all mill-type motors. I feel convinced, after an operating experience of some seven years, that we did the right thing to put in low-voltage D.C. rather than straight A.C.

The accommodation of cables did not present any great problem, and I am sure that so far as the space taken up by the motors, contactor panels, and resistances is concerned, this is no greater with D.C. than it would have been with straight A.C., and is probably less.

Mr. **H. C. Loving**: There is no doubt that A.C. can be used satisfactorily throughout steelworks for cranes whose duty is suitable for it, but D.C. is suitable for all of them and much better for many.

In 1919 we put in at Penistone half-a-dozen 60-ton open-hearth furnaces with a couple of overhead chargers and the charger makers advised us to use D.C. motors throughout, including a 35-h.p. D.C. motor on the travel motion. Our electrician was keen on A.C., and advised it throughout the new melting plant, and so A.C. was adopted.

We ran quite satisfactorily for a time, but after three or four years scrap supplies got much lighter and we had more than twice the number of boxes to a charge. We then began to have serious trouble with the charger travel motors owing to the high speed of working and the frequent reversals. We put in a 50-h.p. motor in place of the 35-h.p., but it heated up, and then a 75-h.p. Maxtorque motor was installed, but with no better results.

The charger makers informed us that at Consett, with similar furnaces, similar scrap, and similar chargers, but with D.C. motors, no trouble was experienced and they suggested a visit. This visit to Consett was made and the makers' claims were confirmed. Consett had been working for nine or ten years with 35-h.p. D.C. travel motors without any trouble at all.

The Penistone plant closed down in 1930, and those chargers were sent to Sheffield in 1932 for use in a new melting shop at Vickers works. The motors remain A.C. with a standard 50-h.p. slip-ring motor on the travel motion, and roller bearings were fitted to the travel axles. The roller bearings have given some trouble and the 50-h.p. motors are to some extent overloaded. The slip-ring surfaces frequently show signs of blistering, but have been able to carry on without serious trouble for the past 14 years.

The conditions at Vickers works are much less severe than at Penistone, there being fewer furnaces and much heavier scrap. Had the same light scrap been used as at Penistone, with consequent rapid working, similar trouble would no doubt have been experienced and certainly the use of 35-h.p. D.C. travel motors would have been a much better solution.

Incidentally, the 100-ton ladle cranes were A.C. and never gave us any serious trouble, but I do not say that D.C. would not have been better. Whether A.C. is suitable or not is not a matter of heavy or light cranes, but of the type of duty they have to perform. The safest procedure is to use D.C. on all steelworks cranes, and especially on handling machines of all types.

Mr. **A. C. Jack** (Messrs. Stewarts and Lloyds, Ltd.): I have been connected with steelworks for thirty-five years, and I am in favour of D.C. I had two overhead 100-ton D.C. cranes, and in twenty-one years' service only one armature has been changed. Mr. Russell Taylor wants to know whether D.C. or A.C. coppers wear quicker on contactors. I would say that there is not much difference. We have tried two or three different types of alloy metal, and we find that we get the best results with pure electrolytic copper. Mr. Grove said that he was instrumental in putting in a steelworks not long ago which was all A.C but if he were to come back to it now he would see that a number of his A.C. motors have now been changed and made D.C.

Mr. **W. Redfern** (The Lancashire Steel Corporation, Ltd.): The discussion which has gone on since I last spoke has simply tended to confirm what I said then, that there is no further argument as to which is best, although, as certain speakers have said, there are some applications where A.C. drive may be cheaper in first cost. That is the only advantage.

Mr. Russell Taylor and others have asked for information with regard to the life of contacts on

A.C. and D.C. contactors. I have had experience with both for many years, and, as has been pointed out, owing to modern developments in the design of blow-outs and so on, the matter is not worth arguing or worrying about, because contacts cost so little, they last so long, and take such a short time to change.

Mr. **A. E. Horrocks** (Messrs. Craven Brothers Crane Division, Ltd.): Speaking from the point of view of a crane maker, I would say that one goes to a job with an open mind, prepared to do what the customer wants. If he buys current from the grid and has only a small establishment he must have A.C.; you may be a D.C. enthusiast but you have to give him an A.C. crane. The value of your crane can be judged by what you hear about it afterwards; on some occasions customers come back to complain.

I agree with Mr. Russell Taylor's suggestion of the D.C. crane operated from A.C. supply with rectifiers.

There is a point which I often use when talking to a customer: If you have a crane required for production and have D.C. and A.C. equally available, if an A.C. crane is installed, to get equal output you must have a 40% increase in speed and horse-power as against D.C.

We had a case of high-speed travel gear on a crane in a steelworks where they used A.C. We put in a very big motor but it had not been running for long before they began to complain of overheating and made us change it, installing a motor of more than double the capacity. Soon afterwards they asked us to take this out because it got just as hot as the original machine. The fact of the matter was it was not the motor loading but the number of starts per hour which heated the motor up.

Personally I am in favour of D.C. and I am very pleased to hear my opinion confirmed today that D.C. should be used on steelworks and heavy-duty cranes.

When we carried out our extension in Manchester in 1912 we bought A.C. current from bulk supply at 6,000 V., and transformed down, and for cranes and all the variable-speed machine-tool drives in the works we converted to D.C., using A.C. on constant-speed machines and line-shafting.

Mr. **E. Edwards** (Messrs. Dorman, Long & Co., Ltd.): I have been interested in the application of electricity to steelworks for over twenty-five years, and this controversy of A.C. *versus* D.C. has always been a live one. From the opinions expressed by most of the speakers it looks as if it is now well on the way to settlement. The firm with which I am connected is largely committed to A.C. distribution and A.C. cranes, and after twenty-five years' experience I can say that we have had excellent service from A.C. motors even on heavy duty such as ladle cranes, and we have had no reason to complain in any way of their performance. All the same, if we were starting again, I feel that D.C. motors would be favourably considered for heavy steelworks duty. To this extent I agree with the arguments advanced this afternoon, and I feel that the characteristics of the D.C. motor are so ideal for crane duties that they encourage its use in spite of recent developments in A.C. control, etc.

I am very interested in Mr. Grove's point. He said that there are applications for A.C. and for D.C. cranes. I feel that either is perfectly satisfactory for certain applications and the choice will depend on the geographical layout of the plant. We know that the primary distribution in a steelworks is A.C., and that to get D.C. we must have special converting plant. The question is whether it is necessary to instal this to give D.C. supply on all cranes. I suggest that for heavy steelworks duty D.C. is most suitable, but that for ordinary applications A.C. is quite satisfactory.

On the question of A.C. contactors we have found that the D.C.-operated type are superior, and have installed small rectifiers for them.

I am very interested in Mr. Parker's arguments about the use of Ward-Leonard sets on ore bridges, and I shall be still more interested to know the results if the scheme to which he referred is proceeded with. His proposal to use 3,000-V., A.C. on the main crane collectors seems a startling innovation; difficulties of adequate guarding, etc., do not appear to be insuperable, and the reduction in the current capacity required is of great advantage.

Mr. **H. H. Broughton** (Cheam, Surrey): If Mr. Rothera's chief objections can be upheld, it is difficult to understand why A.C. motors are used in steelworks; but if the condemnation is intended to refer principally to the use of A.C. motors on cranes, many of the objections are well founded. I often wonder why steelworks engineers continue to sanction the use of ill-designed Weston-type load brakes on cranes intended for heavy lowering duty. Several speakers have referred to the highly objectionable 'chattering' of A.C. contactors and, as far as I am aware, the only known method of eliminating chatter is to arrange for all operating coils to be energized by direct current drawn from a metal rectifier which forms an integral part of the control panel.

One speaker has advocated the use of 3,300-V. cross-wires, but has failed to give acceptable reasons for the recommendation. As every crane designer knows, a voltage of 500 is ample and the use of a lower voltage is desirable. It will be helpful if more information can be given on the sliding contacts that have been proposed, and on the method of earthing the man trolley and bridge The safety of the crane operators in such cases is a matter on which the views of the Factory Department of the Ministry of Labour are likely to have some weight.

Mr. Baker has commented on the rating of motors on a known cycle of operations. The hoisting cycle of an ore bridge is clearly defined but not so the racking, or traversing. In a given plant the racking distance is liable to vary from 50 ft., or thereabouts, to several hundred feet, and the rating of the racking motor is determined by the most severe sustained operating conditions. In a modern man-trolley bridge-type unloader, racking speeds of 1,500 ft./min. are attained, and the usual acceleration of the man trolley is $1\frac{1}{2}$ to $2\frac{1}{2}$ ft./sec./sec. For short trips the free-running speed is figured low and the accelerating rate high ; while for long trips the speed is higher and acceleration lower. On a given bridge the values to be used are determined by the average length of trip, and the principles underlying the estimation of the correct gear ratio are well understood by designers. In fixing the rating of motors, irrespective of the motion, care has to be taken to make provision for the heating effect of electric braking.

Mr. Parker has referred to a number of ore bridges which are being designed, and for which it is proposed to use the Ward-Leonard system of control. The reasons for the novel proposal have not been given, but it is said that the equipment will increase the weight of the man trolley by about 10%. The heavier trolley will necessitate a heavier bridge, and all to no purpose. To my knowledge, before 1914, the application of Ward-Leonard control to coal and ore bridges was considered and rejected. Had the system possessed any advantages over straight control, a reasonable assumption is that it would have been used more than 30 years ago in the United States by the engineers to whom we owe not only the ore bridge but also the Ward-Leonard system. For certain special types of cranes—other than coal and ore bridges—where high-precision control is necessary, the use of Ward-Leonard control is desirable. Other applications of Ward-Leonard control to mechanical-handling plant are the skip hoist and heavy-duty mine hoist.

In deciding upon the system of control to be adopted it has to be remembered that the precision which characterizes the Ward-Leonard system is not required for coal and ore handling. Moreover, to obtain high operating speeds and large outputs, it is necessary to minimize weights. On this account it is impossible to justify a 10% increase in the weight of the man trolley by providing equipment and accessories of no value from a tonnage-output standpoint.

Bearing in mind that the function of an ore bridge is the economical handling of large tonnages of ore, I think that the feasibility of augmenting the ratio of net load to grab weight should be thoroughly investigated. In my submission the use of alloy steels for the construction of the man trolley and grab is a worth-while investment.

The choice usually rests between straight D.C. and A.C. equipments. For grabbing duty simple A.C. control and counter-torque braking can be relied upon to give good results ; but for a given tonnage output A.C. motors will be 25 to 33% larger than D.C. motors, and on this account D.C. equipments are generally favoured. In both cases the service conditions are such that the use of mill-type motors is imperative and contactor control is necessary for all motions with the exception of the turntable (slewing) motion.

Some years ago I had occasion to examine many of the ore- and coal-handling bridges in the United States of America, as well as a number of installations on the Continent, and the following particulars and data may be of interest.

The motors on a typical heavy-duty man-trolley bridge may have an aggregate rating of about 700 h.p. For three or four such bridges working in concert on a tonnage basis, that is, when the remuneration of the operators depends on the tonnage handled, the R.M.S. value of the demand will be in the neighbourhood of 1,200 to 1,400 h.p., which figure determines the size of the sub-station. The machinery installed in the sub-station is designed to take care of the momentary heavy peaks, and in some cases flywheel load-equalizers are used. With a well-designed plant a total energy consumption of 0·4 to 0·5 kWh. per ton of ore handled is usual.

I doubt the wisdom of attempting to combine A.C. and D.C. motors on a crane. For well-known reasons both kinds of motor are to be found in a steelworks, but these reasons do not apply to crane equipments. Discussion on A.C. *versus* D.C. for steelworks cranes cannot be expected to be very profitable because many years ago, after a searching investigation, it was decided that D.C. was superior to A.C. for such duty and the advent of the mercury-arc rectifier has strengthened the

case in favour of D.C. It is satisfactory to note that several speakers regard the decision as a sound one.

I have been expecting to hear of developments in A.C. control ; apparently the only development is in connection with the braking of induction motors. Mr. Russell Taylor is not correct in giving credit to American engineers for the use of a variable resistance in one phase. Actually, the invention is due to the Siemens-Schuckert Company and has been extensively used by them, chiefly in conjunction with other braking systems. The main objection to the method is that it is liable to give rise to excessive heating of the motor unless the frame size is increased. I think it not unlikely that much wider use will be made of 'D.C. injection' in the braking of induction motors. A recent promising A.C. hoisting-control development is known as reactor control. In this system the degree of unbalance applied to the motor terminals is varied as a function of the motor speed, and the relationship is adjusted to give the various operating points required.

It has to be recognized that the function of the electro-mechanical brake on the hoisting motion of a crane is that of holding the mechanism at rest, not that of retarding the motion. Most of the brakes of this kind, and of the Weston type, have neither heat-storage capacity nor cooling surface sufficient for dissipating the energy of motion. The substitution of electro-hydraulic systems and motor magnets for solenoid brake-releasing electromagnets is an encouraging and overdue development.

Mr. **J. Parker** : I should like to refer to the question of voltage. My own opinion is that all voltages are dangerous, whatever the degree, and in proposing the use of a voltage somewhat higher than that to which we are accustomed, the collector gear would naturally be arranged out of reach and safely located.

With regard to space on the man trolley, we have drawings showing a very nice arrangement of the motor-generator set on the trolley, and I am sorry I have not brought these with me to show Mr. Broughton. It is perfectly feasible. The fact that the majority of the ore bridges in the U.S.A. are D.C. and seem to go on being D.C., is to me, a very good reason why we should change to A.C.

Mr. **L. Rothera** : I am a little disappointed in one sense with the discussion which has taken place. I backed up D.C. in the hope that someone of a younger generation would tell us that we were thirty-five years behind the times, and I hoped that there would be a much better case put up for some of the later developments in A.C. In spite of that, I remain a convinced upholder of D.C.

There are one or two points, however, with which I should like to deal. I spoke of danger. I think it is fully accepted now that, whilst D.C. can be dangerous it is not so dangerous as A.C., and the real reason is that a shock from D.C. knocks you off, and a shock from A.C. holds you on. That is really the difference between the two. Both are fatal at 460 V. if maintained.

The question of power-factor improvement was touched on, and a synchronous motor was suggested as one of the methods of improving the power factor. That, however, is expensive and means additional running machinery. I think in most cases one would now instal condensers for improving the power factor. If you have converting machinery or drives which will enable you to use a synchronous motor, there is, however, a good deal to be said for using it.

With regard to long-travel brakes, on most of our cranes in the marshalling yard, and so on, we have a foot brake which it is difficult to keep in action, and the whole of the control is done by plugging, as this is quickest and simplest from the driver's point of view. It puts the biggest strain on the machine, but that is how it is done. We usually have a shunt brake in addition which comes into operation only if the supply fails.

I am very interested in Mr. Parker's suggestions for ore bridges, but Mr. Broughton dealt with the point I was going to make on the question of the diversity factor. If you put a motor-generator set on the crane it has to be the full capacity of the crane, whilst if you have three or four ore bridges you only want 50% more on your converting station, and that would go very far towards meeting the extra cost. If Mr. Parker is running 3,300 V. on to the ore bridge he will presumably need a transformer as well, because he did not suggest 3,300-V. motors.

The only other point is this question of voltage. We have up to the present standardized on 230 V., as I think most people have, but it is now many years since I installed an equipment at 500 V. in a steelworks, and from the information that I have been able to gather there have been no complaints of high maintenance or other troubles due to that. If we were installing new equipment today we should certainly move up to 460 V., which goes far to overcome the difficulties and costs of transmission, particularly when you couple with it the introduction of localized rectifier sub-stations with incoming high-tension supply.

Mr. **W. Redfern** (The Lancashire Steel Corporation, Ltd.) : I have been using 500 V., D.C.,

on cranes and other applications for over 30 years, and can take my mind back to the time when we were operating all our rolling-mill auxiliaries with ordinary drum-type controllers. Owing to the destructive effect of arcs in the controllers we had to change the finger tips and liner tips about twice a week to keep the rolling mills working. We have now practically eliminated all those troubles, which used to occur at controllers and motor brush-gear, by the use of magnetic-operated contactor control gear on all rolling-mill auxiliaries. On cranes up to 50 h.p., *i.e.*, 75 amp. at 500 V., we are using cam-operated contactor drum-type controllers to avoid the small wiring and auxiliary contacts associated with magnetic contactors. We are compelled to use magnetic contactors on machines above 50 h.p. because cam-operated contactor controllers are heavy for the driver to operate. As a result of this we have more or less eliminated all the serious troubles we experienced in the past.

Mr. **G. W. Grossmith** (Messrs. Strachen and Henshaw, Ltd.): I would like to comment on Mr. Broughton's remark that Ward-Leonard drive, if advantageous, would have been adopted by American engineers thirty years ago. The implication does not do justice to British engineers, who in fact do not always wait to see what others are doing. A few years ago I was concerned with negotiating a contract for South Africa in which we had to lift and discharge very heavy rail wagons, weighing 90 tons or so, at the rate of 30 per hour, the operating motors being of 550 and 350 h.p. The Chief Engineer of the South African Railways and his staff told me, when we were discussing the proposals, that the American firms tendering for the plant had asserted that it was impossible to build such a large fast-working tippler to run on other than D.C. or by Ward-Leonard drive. A.C. supply only was available, but I was able to demonstrate that by the adoption of new mechanical features it was possible to operate the machine by A.C. just as satisfactorily as by D.C., and at less cost. The contract came to this country, and when installed gave complete satisfaction.

Mr. **P. F. Grove** (Messrs. John Miles & Partners (London), Ltd.) wrote: The supply to the Fontana cranes is A.C., but Ward-Leonard control is employed only on the hoist. The system is, however, flexible and can be adapted for A.C. or D.C. supply on other applications. On A.C. the hoist may have a motor generator and the remaining drives slip-ring induction motors. In some cases, where only one motion at a time is permissible, a single motor generator can be connected alternatively to them. In other cases where two or more operations must be carried out together, it is practical to combine several generators on the motor-generator set. The set includes a constant-potential exciter and a cross-flux exciter, but when the main supply is D.C. the cross-flux exciter only is used, the constant-voltage exciter being obtained from the D.C. power supply.

Mr. **J. Parker**: The cranes referred to by Mr. Grove were specially built for the construction of the Fontana Dam. The two cranes were mainly employed for hoisting buckets of concrete of 4 cu. yd. from the mixing plants to the Dam, but were also used on jobbing work inherent in such construction. Each hoist drive was rated at about 200 h.p., the total lift was about 300 ft., and the overhang of each cantilever about 130 ft.

High operating speeds and accurate control of hoisting and trolley travelling were essential, and Ward-Leonard control was applied to each motion. Each motion had a separate motor-generator set with induction-motor drive, and the traverse motor-generator set had a flywheel.

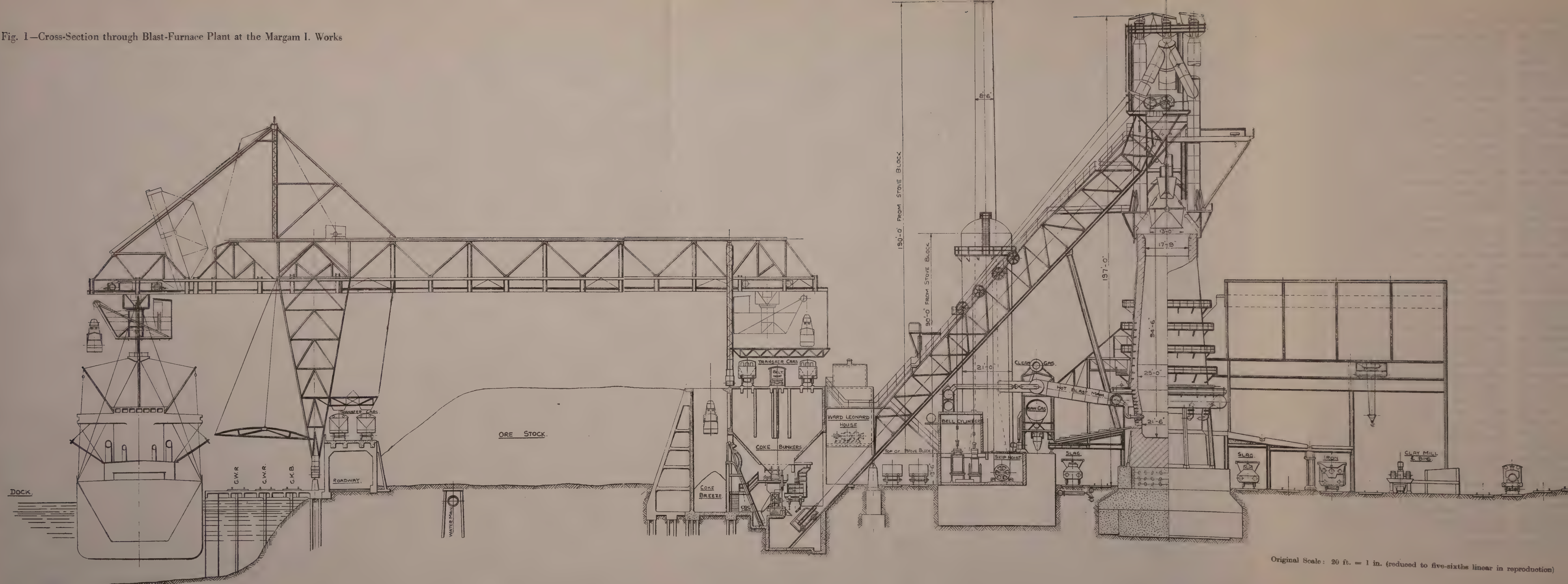
Cross-flux exciter control was used on the hoists to limit the hoist-motor torque, since the cranes were of the cantilever-gantry type and it was essential to protect against excessive overturning moments.

The electrical equipment was designed by the General Electric Company, Schenectady.

Mr. **Russell Taylor**: I should like to give my experience of a case where there was a very large forging crane of about 250 tons. This crane had to have the usual forging chain on it, and the problem was to hoist it and lower it 6 in., the hoisting speed being 12 ft./min. The hoisting of 6 in. was a complete cycle, ten to the minute. The crane had been put in with an amplidyne on the hoist, and the problem was whether this high-duty cycle could be done on straight D.C. It was put up to me, and the customer wanted a guarantee. I said that I was an engineer and not a salesman, but I thought that I could get somewhere near it. Two 150-h.p. motors were used on the hoist, and a perfectly normal two-motor dynamic lowering controller was put on, and on the test I got ten complete cycles in 67 sec., which was not quite ten a minute. I said: "Give me another try, and I think that I can do it," but they said "No, you have given us 8 in. instead of 6 in." That is a case where the amplidyne was put in to obtain high acceleration, and it was done with straight D.C.

(*End of the discussion*)

Fig. 1—Cross-Section through Blast-Furnace Plant at the Margam I. Works



Original Scale: 20 ft. = 1 in. (reduced to five-sixths linear in reproduction)

To face p. 289]

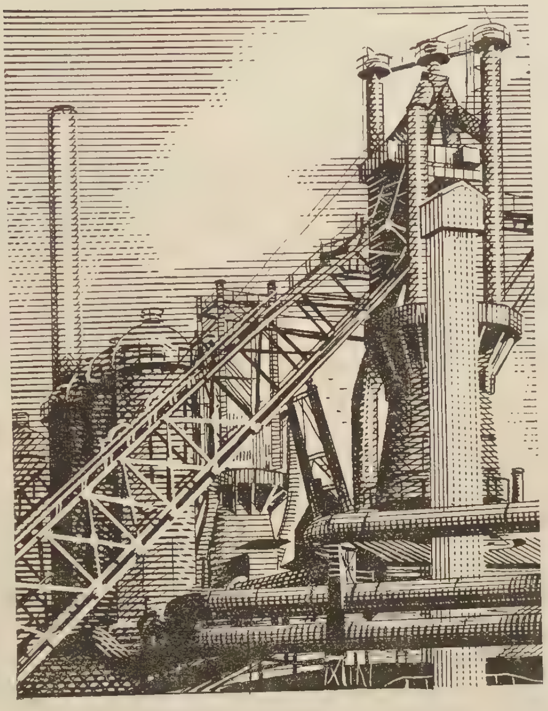
No. 1 Blast-Furnace at the Margam I Works

MESSRS. GUEST KEEN BALDWINS IRON AND STEEL CO., LTD.*

It was decided in 1944 to rebuild No. 1 Blast-Furnace at Margam to a hearth diameter of 21 ft. 6 in. to replace the then existing furnace of 14 ft. 0 in. dia. Figure 1 shows a cross-section of the plant through the new furnace.

Part of the original foundation was left in. This was capped and enlarged to carry the new furnace, giving a bearing pressure on the ballast of approximately 2 tons/sq. ft. The new furnace was blown-in on 8th August, 1946.

The design of the furnace follows the most modern practice and has a rated capacity of 5000 tons per week. The principal dimensions are as follows:



View of No. 1 Blast-Furnace showing passenger hoist

Hearth diameter	21 ft. 6 in.
Bosh diameter	25 ft. 0 in.
Stockline diameter	17 ft. 9 in.
Large bell diameter	13 ft. 0 in.
Annular space bell–stockline ...	2 ft. 4½ in.
Bottom of hearth to tap-hole ...	1 ft. 6 in.
Tap-hole to slag notch	4 ft. 0 in.
Slag notch to tuyeres	3 ft. 3 in.
Tuyeres to stockline	73 ft. 6 in.
Stockline to top decking	12 ft. 3 in.
No. of tuyeres	14.
Bosh angle	80° 32′ 16″
Height of bosh (sloping section) ...	10 ft. 6 in.
Height of bosh (parallel section) ...	10 ft. 0 in.
Inwall batter	0·989 in. in 12 in.
Volume, tuyeres to stockline ...	27,716 cu. ft.

The furnace lining weighs 1833 tons, the alumina content of the refractories being:

Hearth ...	40–42%
Bosh ...	40–42%
Inwall ...	34–36%
Throat ...	34–36%

The furnace stack and tuyere belt is cooled by inserted water-cooled copper plates. The hearth jacket, of rolled steel plate backed with 12 in. of rammed coke and tar, is cascade cooled. The total quantity of water used for cooling purposes is approximately 250,000 gal./hr.; this quantity covers tuyeres, tuyere coolers, plate coolers, and cascade.

The various materials, excluding coke, are collected from the bunkers by means of an Atlas scale car through compressed-air-operated bin gates. The scale car has two hoppers of 160 cu. ft. capacity which discharge when registered, into the blast-furnace charging skips. The scale-car doors are operated by compressed air from a portable air compressor on board.

The new furnace is charged by double skip, each skip being of 160 cu. ft. capacity capable of carrying 4500 lb. of coke, discharging into a McKee distributor top.

All bearings and wearing parts, including the McKee top, are lubricated by the Farval system from a central pump situated in the hoist engine house at ground level.

* Received 22nd November, 1946.

Access to the furnace-top platform for inspection and repairs is by means of a passenger hoist from ground level.

The furnace-hoist engine house which houses the main hoist, bell cylinders, and depth-rod winches, also houses in a separate compartment, the electrical control panels. The whole building is air conditioned. The full sequence of charging events is interlocked, and the only operator between the service bunkers and the tap-hole is the scale-car driver.

The coke bunker is situated on the furnace centre-line and discharges to the right and left skips by means of C. C. Larssens grizzly screens which also act as gates and conveyors. The screens are designed to extract breeze up to $\frac{3}{4}$-in. ring. The disposal of the breeze is by an automatic skip which places breeze within reach of the transporter cranes, which in turn load it into railway trucks. The C. C. Larssens screens discharge into weighing and/or measuring hoppers which have a capacity of 4500 lb. of coke.

The charging sequence of O O C C O O C C is usually followed. A typical ore burden consists of the following materials :

Obregon	33·4%
Kiruna	25%
Spathic	16·7%
Oxford	12·5%
Agglomerate	8·3%
Mai-sur-Orne	4·1%
	100·0%

The normal blast volume for the furnace is 50,000 cu. ft./min. with a maximum of 55,000 cu. ft./min. When blowing 46,000 cu. ft./min. at 16–17 lb./sq. in. the make is about 4600 tons. The cold-blast main from the blower to the stoves is 48 in. diameter.

The furnace is served by four existing stoves operated in pairs, two on gas and two on blast. These are 21 ft. dia. by 96 ft. 7 in. high with 'Hotspur' fillings giving 129,280 sq. ft. of heating surface. The method of stove changing is such that only one stove is changed at one time. Stoves are normally on blast for 6 hours but a hot stove is brought into operation every 3 hours. They are fitted with Steinbart burners with automatic air/gas ratio control. Each stove takes 17–18% of the furnace make of gas, and the hot-blast temperature is automatically controlled at about 1000° F.

Compressed-air drills and augers are used for opening the tap-hole, and a column-mounted Brosius mud gun of 9 cu. ft. capacity is used for plugging the hole. The iron-runners are laid with a minimum fall of 0·625 in./ft. and are arranged for the filling of 4 ladles if necessary.

The molten iron is handled in Kling ladles of 75 tons capacity when newly lined, increasing to 96 tons when worn. The maximum total weight of the ladle, carriage, and contents being 153½ tons. The molten iron is transferred by means of the above ladles, either to the mixer situated in the melting shop or to a double-strand pig-casting machine capable of handling 2 tons/min.

A typical pig-iron analysis is as follows :

Si, %	S, %	Ph, %	Mn, %
0·5	0·05	0·38	1·2

There are two slag notches, one 51 degrees from the tap-hole, and one almost diametrically opposite. The minimum fall in the slag-runners is 0·75 in./ft., with 6 spouts for normal slag, and 5 spouts for roughing slag.

The slag is at present handled in 220-cu. ft. chain-tipped slag ladles. It is proposed to use air- or steam-tipped ladles of 400 cu. ft. capacity in the future. The slag volume approximates 10 cwt. per ton of pig iron, and the slag is tipped into a tidal area about 1 mile's distance from the furnaces.

The top-gas temperature at the furnace is about 350° F. The gas passes through a dustcatcher and cyclone in series ; the gas volume being approximately 3,000,000 cu. ft./hr. for the output stated above.

The average amount of dust loss with the present burden and blast volume is approximately 90 lb. per ton of pig iron—this includes all dust from the dustcatcher, crude-gas main, and gas cleaning plant. The gas analysis when blowing at 30,000–35,000 cu. ft./min. is :

CO_2, %	CO, %	H_2, %	N_2, %
13·8–15·2	25·2–26·8	2·4–2·7	56·7–57·2

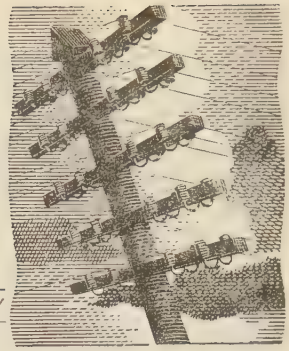
	B.Th.U./cu. ft. Net	Gross
Calorific value	86–93	88–93

When blowing at 46,000 cu. ft./min., the following is a typical analysis :

CO_2 %	CO, %	H_2,%	N_2,%
12·9	27	3·1	57

The gas in this example had a calorific value of 94 B.Th.U./cu. ft. net and 96 gross.

N E W S



ANNOUNCEMENTS AND NEWS OF SCIENCE AND INDUSTRY

THE IRON AND STEEL INSTITUTE

Joint Conference on Desulphurization and Dephosphorization, with particular reference to Cupola Iron

Further details are announced of the meeting, organized by the Institute of British Foundrymen in collaboration with the Iron and Steel Institute, to discuss papers on this subject.

The meeting will be held at 4, Grosvenor Gardens, London, S.W.1, on Wednesday, 19th March, 1947, and will be divided into two sessions. Mr. D. H. Wood, President of the Institute of British Foundrymen, will preside at the morning session from 10.30 a.m. to 12.45 p.m., when Mr. L. W. Bolton (Chairman of the Basic Cupola Sub-Committee of the Technical Council of the Institute of British Foundrymen) will introduce the First and Second Reports on the Basic Cupola Iron.

After a Buffet Luncheon the afternoon session will commence at 2.15 p.m. when Mr. Bolton will introduce a report on desulphurization of cupola iron and pig iron in basic lined ladles, with an appendix on dephosphorization.

The discussion on both papers will be opened by Mr. F. L. Robertson.

The First Report of the Basic Cupola Committee has been published as Paper No. 803 in the Proceedings of the Institute of British Foundrymen, 1943/44, Volume 37. The Second Report was issued as Paper No. 858 in pre-print form in May, 1946. The report on desulphurization has not yet been published, but copies of all papers will be available to members attending the meeting.

A notice about the Conference has been circulated to Members with a request to return the completed forms as soon as possible.

Special Summer Meeting in Zurich, 1947

A circular (C.693) has now been sent to all Members outlining the programme for the proposed special Summer meeting in Switzerland in July, 1947, which is to be held with the collaboration of the Swiss steel and engineering industries.

The meeting will take place in Zurich on the 10th and 11th July, and will be preceded by a Reception by the Municipality of Zurich on the evening of July 9th. Following the end of the discussion of papers, visits to works and excursions are being arranged for the four days from 12th to 15th July. This ends the official programme for the special meeting, but arrangements are being made for a five-day excursion through Switzerland, or an alternative five-day excursion to Geneva, with visits to local works of interest during the period from 16th to 21st July.

During the afternoons, free of the discussion of papers, a series of interesting visits to local factories and sightseeing tours has been arranged for Members and ladies, and there will be a number of social activities provided in the evenings.

A number of special papers are being prepared by Swiss and English authors for presentation and discussion at the meetings, which will take place in the Eidgenossische Technische Hochschule by invitation of the President of the Schweizerisches Schulrat. The whole of the papers, if received in time, will be published in the June issue of the *Journal*.

Special Note—Members wishing to receive additional information have been requested to return, by 15th February, the reply form attached to the circular. Will Members, who have not already done so, please return the forms without delay.

Joint Library

TRANSACTIONS OF THE AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS

The Library requires Volumes 148 and 157 to complete its set of the "*Transactions of the American Institute of Mining and Metallurgical Engineers.*" These are out of print and unobtainable in the second-hand market. Would any Member having spare copies of these Volumes available for disposal please communicate with the Librarian.

Staff Biography No. 2

Mr. Richard Elsdon, Librarian, who celebrates his 60th anniversary this month, was born in London and was educated at the Stephens Higher Grade School and the Technical Institute, Westminster.

He joined the Staff of the Institute in 1904 under the secretaryship of Bennett H. Brough. He was promoted to Library Assistant in 1907 and was appointed Librarian in 1908. During his long association with the Library he has introduced several developments including the loan of volumes to Members, which was sanctioned by the Council in 1914, and the compilation of bibliographies, some of which form the Bibliographical Series published by the Institute. He also began and developed the Information Department which is operated in conjunction with the Library. In addition to his many duties as Librarian Mr. Elsdon has served as an abstractor for the *Journal* and compiled the Indexes for many years.

When the British Iron Trades Association ceased to function in 1915 the work of collecting statistics of the iron trade was undertaken by the Iron, Steel and Allied Trades Federation, and Mr. Elsdon was asked to undertake the compilation of several statistical reports under the supervision of George C. Lloyd who acted as Statistical Secretary to the Federation. This work was afterwards taken over by the National Federation of Iron and Steel Manufacturers, now the British Iron and Steel Federation.

During the first World War Mr. Elsdon served in the Essex Regiment and the Royal Inniskilling Fusiliers and was severely wounded at Langemarck during the 3rd Battle of Ypres in 1917. During the last war he was in the Home Guard.

In 1937 when the Libraries of the Institute of Metals and the Iron and Steel Institute were amalgamated he was appointed Librarian of the Joint Library.

Andrew Carnegie Scholarships

As the result of a generous grant by Mr. Andrew Carnegie, Vice-President of the Institute in 1901, an award of a Gold or Silver Medal and Research Scholarships have been available annually. Candidates, who must be under thirty-five years of age, must apply on a special form to the Secretary of the Institute.

The scheme enables students who have passed through a college curriculum or have been trained in industrial establishments, to conduct researches in the metallurgy of iron and steel and allied subjects. The research may be carried out at any place selected, provided that it is properly equipped for the prosecution of metallurgical investigation.

The award of a Scholarship is for one year, but the Council may, at their discretion, renew the Scholarship for a further period. The value of a Carnegie Scholarship will not normally exceed £250.

The results of the research must be communicated to the Iron and Steel Institute in the form of a paper, and the Andrew Carnegie Medal is awarded to the author of the best paper in each year if it is considered by the Council to be of sufficient merit.

THE BRITISH IRON AND STEEL RESEARCH ASSOCIATION

Steel Castings Research Committee

As announced in the January *Journal*, a meeting has been arranged with the Association and the Iron and Steel Institute to present and discuss the reports of the Sub-Committee of the Steel Castings Research Committee. Members of the Institute of British Foundrymen are cordially invited to be present.

The meeting will be held at 4, Grosvenor Gardens, London, S.W.1, on Thursday, 20th March, 1947. Professor J. H. Andrew has been asked to preside.

The morning session, from 10.30 a.m. to 12.45 p.m., will be devoted to the discussion of the First Report of the Converter Refractories Sub-Committee which will be introduced by its Chairman, Dr. R. J. Sarjant.

During the afternoon session, from 2.15 p.m. to 4.30 p.m., the First Report of the Side-Blown Converter Practice Sub-Committee will be introduced by Dr. T. P. Colclough, Chairman of the Sub-Committee. In the interval between the two sessions a Buffet Luncheon will be served.

The paper to be presented at the first session appears in this issue of the *Journal* (*see* pages 253

to 269. The paper which will be presented at the afternoon session was published in the January *Journal* (*see* pages 33 to 50). A notice has been circulated to members and completed reply forms should be returned as soon as possible.

THE IRON AND STEEL ENGINEERS GROUP

The **Third Meeting** of the group will be held at 4, Grosvenor Gardens, London, S.W.1, on Wednesday, 26th February, 1947, when three papers on "*The Blast Furnace of Today*" will be presented and discussed. These papers were published in the January *Journal* (*see* pages 107 to 140).

Joint Meeting with the South Wales Branch of the Institute of Mechanical Engineers

A joint meeting of the South Wales Branch of the Institution of Mechanical Engineers and the Iron and Steel Engineers Group will be held at the Mackworth Hotel, Swansea, on Tuesday, 4th March, 1947, at 6 p.m. A lecture on "*Some Possible Future Developments in Iron and Steel Manufacture in Great Britain*" will be given by Mr. W. F. Cartwright.

Members of the Swansea and District Metallurgical Society are cordially invited to be present and join in the discussion.

NEWS OF MEMBERS

Recent Appointments

▸ Sir CHARLES BRUCE GARDNER has been appointed Chairman of John Lysaght Ltd.

▸ Col. Sir W. CHARLES WRIGHT has retired from the Chairmanship of Guest Keen Baldwins Iron and Steel Co., Ltd., but is remaining on the Board.

▸ Mr. JAMES H. JOLLY has been appointed Chairman of Guest Keen Baldwins Iron and Steel Co. Ltd.

▸ The following members of the Institute have been appointed Executive Directors of Colvilles Ltd. :—

MR. WILLIAM BARR, Chief Metallurgist; MR. THOMAS W. HIND, Chief Mechanical Engineer; MR. LAMBERT ROTHERA, Chief Electrical Engineer; MR. JOHN MCCRACKEN, Works Manager, and MR. ROBERT MARSHALL, Sales Manager.

▸ Mr. EDWARD T. JUDGE has been appointed a Director of Dorman Long and Co., Ltd.

▸ Dr. R. J. SARJANT has resigned from Hadfields Ltd. and has accepted the Chair of Fuel Technology at Sheffield University.

▸ Mr. T. LAND and Mr. H. W. PINDER have resigned from Wm. Jessop and Sons Ltd. Mr. Land has formed the company of T. Land and Sons Ltd. at Colonial Works, 318, Queens Road, Sheffield, 2, dealing in pyrometric equipment and scientific instruments. Mr. Pinder has also commenced business on his own account at 6, Taptonville Road, Sheffield, 10.

Elections

▸ Sir WALTER BENTON JONES has been elected President of the British Tar Confederation.

▸ Mr. ELLIS HUNTER has been re-elected President of the British Iron and Steel Federation for 1947.

▸ Lord RIVERDALE has been elected the First President of the Production Engineering Research Association.

▸ Mr. C. S. GILL has been re-elected Chairman of the British Steel Founders' Association for 1947.

▸ MR. CLYDE E. WILLIAMS has been renominated as President of the American Institute of Mining and Metallurgical Engineers.

Honours

The President and Members of the Council offer their congratulations to the following members :—

▸ Mr. R. A. HACKING of Dorman Long and Co., Ltd., and Dr. L. B. PFEIL of The Mond Nicke Co., Ltd., who have been awarded the O.B.E. in the New Year's Honours.

▸ Mr. J. D. GILL, of Vanadium-Alloys Steel Co., U.S.A., who has received the Honorary Degree of Doctor of Engineering from the University of Missouri.

▸ Mr. LOUGHNAN ST. L. PENDRED, C.B.E., who has been elected a Fellow of the City and Guilds of London Institute.

▸ Dr. J. L. HAUGHTON and his wife (Dr. MARIE GAYLER) of the National Physical Laboratory, who have received the joint award of the Institute of Metals Platinum Medal for 1947.

Obituary

The Council regret to record the deaths of the following Members :—

Dr. J. W. DONALDSON, Honorary Member of Council and President of the West of Scotland Iron and Steel Institute.

Mr. JONES FOSTER-SMITH, Managing Director of The William Smith Owen Engineering Corporation Ltd.

MR. JOHN GETHIN-JONES, B.Sc., Steelworks Manager, W. Gilbertson & Co., Ltd., Glamorgan.

Appreciations of these Members will be given in the March issue of the *Journal*.

CONTRIBUTORS TO THE JOURNAL

Thomas Fairley, Ph.D., M.Sc., F.Inst.P., A.F.R.Ae.S., F.I.M., M.I.E.I.—Assistant Works Manager of the Park Gate Iron and Steel Co., Ltd.

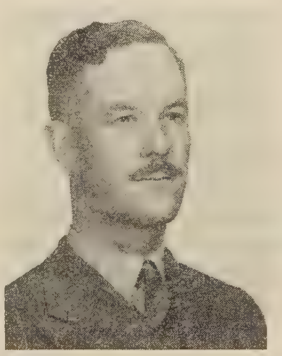
Dr. Fairley received his technical education at Sheffield University from 1927 to 1937. He was awarded an Honours B.Sc. degree in Physics in 1930 and his M.Sc. degree in the following year; after specialising in metallurgy he obtained a Ph.D. degree in 1934. Dr. Fairley was Lecturer at Loughborough College from 1937 until 1940. He then joined the Park Gate Iron and Steel Co., Ltd., as a Metallurgist, and was promoted to his present position as Assistant Works Manager in 1946.

J. E. Hurst, J.P., D.Met.,—Director of Research to The Staveley Coal and Iron Co., Ltd., Nr. Chesterfield, and Technical Director of Bradley and Foster Ltd., Darlaston. He is a Past-President of the Institute of British Foundrymen, and also Past-President of the Staffordshire Iron and Steel Institute; a Member of the Institution of Mechanical Engineers, and a Justice of the Peace for the City of the County of Lichfield. He has been intimately associated with the development of the centrifugal casting process in Great Britain, particularly in its application to special cast irons for high-duty purposes. In 1918 and 1919 he was Editor of the Foundry Trade Journal.

Dr. Hurst has made numerous and original contributions to the Iron and Steel Institute, of which he is a Member and a Carnegie Research Scholar, and also to various other societies and technical bodies. In 1932 he was awarded the Oliver Stubbs Gold Medal, of the Institute of British Foundrymen, in recognition of his outstanding contributions to the metallurgy of cast iron, particularly in the form of new alloys. In 1938 he also received, from that Institute, the E. J. Fox Gold Medal. He has also conducted extensive research into the possibilities of hardening and tempering various cast irons and alloy cast irons. Results of some of his investigations were presented to the Institute of British Foundrymen in a series of Papers commencing in 1930. Dr. Hurst is also a Member of the Council of the British Cast Iron Research Association.

R. V. Riley, Ph.D.—Research Manager at the Staveley-Bradley and Foster Research Laboratories, received his technical education at Leeds University, leaving, in 1938, with a B.Sc. degree (1st Class Honours in Metallurgy). He joined the United Steel Companies Ltd. for a year, but returned to the University to carry out research work for the Alloy Steels Research Committee of the Institute. After receiving his Ph.D. degree he joined the Ministry of Supply as a metallurgist in 1942, and for four years was in charge of research and production at the Government Acid Resisting Silicon Iron Factory at Darlaston. Dr. Riley accepted his present appointment in 1946.

James White, D.Sc., Ph.D., A.R.T.C.—Lecturer in Refractory Materials, Sheffield University. Dr. White was educated at Langholm and Dumfries Academies and Glasgow University. He graduated in 1931 with a first-class Honours degree in Physical Chemistry and obtained his Ph.D. degree in 1935. The degree of D.Sc. was conferred on him at Glasgow University, in 1939.



As a Research Scholar in Metallurgy (D.S.I.R.) from 1931 to 1933 Dr. White continued his metallurgical studies at the Royal Technical College, Glasgow, under Professor Andrew and later Professor Hay, and from 1933 to 1935 was Research Assistant to Professor Hay. He was awarded an Associateship of the Royal Technical College in Metallurgy in 1933 and served at the College from 1935 as a lecturer in Metallurgy until, in 1943, he was appointed Research Technologist at Messrs. General Refractories, Ltd., Worksop. He took up his present appointment in January, 1946.

Dr. White was elected a Member of the Iron and Steel Institute in 1938, having held, in the two previous years, a Carnegie Scholarship. For his paper on "Equilibrium at High Temperatures in Systems containing Iron Oxides" he was awarded the Andrew Carnegie Gold Medal in 1939.

Hugh McKenna Skelly, Ph.D.—Metallurgist at Messrs. Joseph Sankey and Sons, Ltd., Wolverhampton, graduated from Glasgow University in 1938, with honours in Physical Chemistry. He obtained his Ph.D. degree after carrying out metallurgical research at the Royal Technical College, Glasgow, during 1938 to 1941.

During the last war he worked in the Physical Chemical section of the Armament Research Department, Woolwich, on topics relating to the detonation of explosives.

Mr. H. J. Merchant—Metallurgist at Messrs. Commercial Steels and Forge Co., Pty., Ltd., Sydney, N.S.W.

The paper by Mr. Merchant on "The Application of Electrolytic Polishing to Ferrous Metallography" has been based on researches carried out with the aid of an Andrew Carnegie Scholarship awarded to the author in 1942.

Mr. Merchant received his general education at King Charles I School, Kidderminster, and continued his Technical Education in the Metallurgy Department of Birmingham Central Technical School. After serving at the College as a part-time lecturer in Iron and Steel Manufacture he accepted, in 1931, an appointment as Assistant Metallurgist at the Midland Branch Laboratory of Messrs. Baldwins Ltd. In 1941 he was appointed to his present position, and during his journey to Australia he took the opportunity of visiting various steelworks and forges in America.

A. J. Gould, Ph.D., M.I.C.E., M.I.Mech.E., M.I.N.A.—Lecturer at Cambridge University. Educated at the Royal Naval College, Greenwich, and King's College, Cambridge, Dr. Gould served for some years with the Naval Construction Department of the Admiralty. He was appointed Lecturer, and later Professor of Engineering at the University of Rangoon and following this took up his appointment at the University of Cambridge where, for many years, he has worked in collaboration with Dr. Evans upon problems of corrosion.

U. R. Evans, M.A., Sc.D.—Reader in the Science of Metallic Corrosion at the University Chemical Laboratory, Cambridge, Dr. Evans was educated at Marlborough College and received further academic and technical education at King's College, Cambridge, where he obtained his B.A. degree in 1910 and a M.A. degree in 1914. He received the degree of Sc.D. in 1932.

Apart from numerous visits abroad to give lectures by invitation, and service in the Armed Forces during the First World War, Dr. Evans has devoted his time to research on metals at Cambridge University. His work has been applied particularly to corrosion reactions, a subject in which he is a recognized authority and on which he has written numerous books and papers.

Dr. Evans was elected a Member of the Iron and Steel Institute in 1924, and has also been a Vice-President of the Faraday Society and the Cambridge Philosophical Society.

METALLURGICAL FILMS

A request is made to all firms and Industrial Research and Development Organizations who own, or have made, films dealing with Metallurgical Research, or showing some scientific aspect of the production and uses of metals, to communicate with the Education Officer at the Iron and Steel Institute, 4, Grosvenor Gardens, S.W.1, from whom copies of a circular may be obtained setting out the " Particulars of Films " which are particularly required.

On behalf of the Joint Committee on Metallurgical Education, a list of films including brief synopses, and details regarding running time, date of production, source from which obtainable, etc., has already been compiled on the production and uses of iron and steel. A similar list, for the non-ferrous industries, is now in the course of preparation and the intention is eventually to build up a Metallurgical Film Library of suitably graded and appraised films from which a selection can easily be made of those suitable for showing to Colleges, Schools, Apprentice-Trainees or Specialist Scientific Audiences, on any subject.

At present many metallurgical films are scattered around the country and remain in places where their existence is unknown and their circulation accordingly restricted. What is now needed is a Central Library where copies of films can be deposited and in which the booking, hiring charge, dispatching and repair services can be properly handled. In this way, the cost of replacing damaged and worn prints could be covered and the films given the publicity many of them deserve.

STAHL UND EISEN

The publication of this important periodical is now resumed, as from January, 1947.

RHEOLOGY OF SOLIDS

. A conference has been arranged jointly with the Midland Branch of the Institute of Physics and the British Rheologist Club, at the University, Birmingham, on 28th February and 1st March, 1947.

At the first session on Friday, 28th February, at 6 p.m., to be held at the University, Edmund Street, Dr. Orowan will give an introductory address on "*The Mechanism of Phase Transformations in Solids*."

At the second session on Saturday, 1st March, at 10.0 a.m., to be held at the Physics Dept., The University, Edgbaston, Dr. Hugh O'Neill (metals, and Mr. Gurney (non-metals) will speak on the practical aspect of hardness measurement.

THE INSTITUTE OF FUEL

Conference on Pulverized Fuel

A Conference on Pulverized Fuel, organized by the Institute of Fuel, will be held in Harrogate from Tuesday, 3rd, to Friday, 6th June, 1947.

More than fifty papers will be contributed from the Electricity Supply Industry, Water Tube Boiler Makers, the Fuel Research Station, the British Coal Utilization Research Association, National Coal Board, the British Refractories Research Association and others, including overseas organizations. These papers are to be published in book form.

All Members are invited to take part in this Conference, which will also include various social activities. Particulars can be obtained from the Pulverized Fuel Conference Committee, the Institute of Fuel, 18, Devonshire Street, London, S.W.1.

TRANSLATION SERVICE

Since the announcement made in the January issue of the *Journal* (*see* page 144), further translations have been put in hand and the following are now available or in course of preparation.

TRANSLATION AVAILABLE

No. 300 (German.) H. BENNEK, W. KOCH, and W. TOFAUTE: "The Production of Chromium Coatings by Diffusion." (*Stahl und Eisen*, 1944, vol. 64, Apr. 27, pp. 265–270.) (Translated by Mr. W. B. Jones and made available through the courtesy of Tube Investments Ltd.)

TRANSLATIONS IN COURSE OF PREPARATION

(German). W. LUEG and A. POMP: "Tests to Determine and Predict the Drawing Force in the Bright-Drawing of Steel." (*Mitteilungen aus dem Kaiser-Wilhelm-Institut für Eisenforschung*, 1944, vol. 27, No. 4, pp. 43-52.)

(Swedish). S. MÖRTSELL: "Rationalization in Swedish Iron-Ore Dressing." (*Jernkontorets Annaler*, 1946, vol. 146, No. 9, pp. 369-460.)

(Swedish). G. PHRAGMÉN and B. KALLING: "The Effect of Temperature on the Equilibrium between Carbon and Oxygen in Molten Steel and in a Gas Phase." (*Jernkontorets Annaler*, 1939, vol. 123, No. 5, pp. 199–218.)

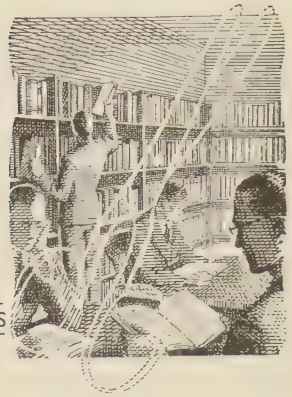
(Swedish). R. TOSTERUD and E. STENFORS: "Investigation of the Wear of Rolls during the Hot-Rolling of Wire and Strip." (*Jernkontorets Annaler*, 1946, vol. 130, No. 6, pp. 213-238.)

(German). H. UNCKEL: "Phenomena in the Pressing of Metal Powders." (*Archiv für das Eisenhüttenwesen*, 1945, vol. 18, Jan.-Feb., pp. 161-167.)

CHARGES FOR COPIES OF TRANSLATIONS.—For the above translations a charge will be made of 10*s*. for the first copy and 5*s*. for each additional copy of the same translation. Requests for translations should be accompanied by a remittance. These translations are not available for loan from the Joint Library.

TRANSLATIONS PREPARED AT MEMBERS' REQUEST.—Members requiring translations of foreign technical papers are invited to communicate with the Secretary, who will ascertain whether the translations can be prepared for inclusion in the series.

ABSTRACTS OF CURRENT LITERATURE *and* BOOK REVIEWS



IRON AND STEEL MANUFACTURE AND RELATED SUBJECTS

CONTENTS

MINERAL RESOURCES

The Geology and Opencast Mining of the Jurassic Ironstones of Great Britain. W. David Evans. (Bulletin of the Institution of Mining and Metallurgy, 1946, Sept.). A detailed description is given of the principal British Jurassic ironstone beds, and the history of their exploitation. A systematic approach to the planning and development of open-cast sites is suggested, and diagrams are shown for its application, with special reference to the Northamptonshire and Lincolnshire ironstones.—C. O.

Mineral Resources of Scotland. M. Macgregor (National Association of Colliery Managers : Iron and Coal Trades Review, 1946, vol. 153, Dec. 13, pp. 1091–1093). Some geological and economic aspects of Scottish mineral resources are discussed briefly. The author states that Scotland has ample reserves of coal, refractory clays, limestone, oil-shale, and sands for industrial purposes, as well as untapped sources of water power.—C. O.

The Geology of the Namwele-Mkomolo Coalfield, Ufipa District : With Notes on Underground Exploration Carried out by the Tanganyika Government. R. B. McConnell. (Bulletin of the Imperial Institute, 1946, vol. 44, July–Sept., pp. 227–251). The Ufipa coalfields lie near the south-western border of Tanganyika Territory between Lake Rukwa and the southern end of Lake Tanganyika ; they consist of two areas of coal-bearing rocks, the Namwele-Mkomolo Coalfield and the Muze Coalfield. The physical features, geological formations, and structure of the first-named field are described.—R. E.

Brown Coals of Victoria. A. B. Edwards. (Proceedings of the Australasian Institute of Mining and Metallurgy : Iron and Coal Trades Review, 1946, vol. 153, Nov. 8, pp. 819–820). There are widespread brown coal deposits in Southern Victoria and five separate coal-bearing areas have been proved. These are chiefly steam or briquetting coals and only thin bands of coking coal of poor quality have so far been found.—R. A. R.

Chromite Deposits of the North Elder Creek Area, Tehama County, California. (United States Department of the Interior, 1946, Geological Survey, Bulletin No. 945-G).—R. A. R.

The Coal Resources of the Re-Incorporated Districts of Eastern Hungary and Transylvania. I. v. Vitális. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1940, vol. 12, pp. 85–106). —R. A. R.

Studies of the Origin of the Marmaros Ore Beds. E. v. Szádeczky-Kardoss. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1940, vol. 12, pp. 107–140).—R. A. R.

Molybdenite Investigations in South-Eastern Alaska. (United States Department of the Interior, 1946, Geological Survey, Bulletin No. 947-B.)—R. A. R.

ORES—MINING AND TREATMENT

Rationalization in Swedish Mining. S. Anestad. (Jernkontorets Annaler, 1946, vol. 130, No. 9, pp. 319–362). (In Swedish). Having indicated the economic and technical conditions which have made rationalization in Swedish mining a matter of great importance, the author discusses the developments taking place in the Swedish iron ore mines. The increasing use of improved drilling machines and of carbide-tipped bits is emphasized. The different mining methods, especially sub-level caving and block caving, are discussed. Mechanical equipment for loading and transport, such as chutes, chute gates, tubs and tub-couplings, mechanical loaders, hoists, and large breakers for use underground, are described. —R. A. R.

Magnetic Separation Tests on the Bog Ores of Bagamér-Nagyletá. J. Finkey. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1940, vol. 12, pp. 10–15). Magnetic separation tests on Hungarian bog ores are described. The ore is very high in moisture and silica, and contains only 21·0–23·6% of iron. By drying, grinding and magnetic separation a concentrate containing 31·7% of iron was obtained.—R. A. R.

A Test to Determine the Lime Abstraction of Iron Ores. E. S. Hertzog. (United States Bureau of Mines, Report of Investigations No. 3929, 1946, Sept.). A description is given of laboratory tests made to determine the capacity of iron ores and associated minerals for abstracting lime from solutions when concentrating ores by the anionic flotation of lime-activated silica. The weight of lime abstracted from solution by one ton of ore amounted to 40 lb. in the case of a hematite, but limonite, magnetite, specular hematite, calcite, and quartz showed very little tendency to abstract lime.—C. O.

Limestone Preparation Plant at Beecroft Quarry. (Iron and Coal Trades Review, 1946, vol. 153, Sept. 20, pp. 483–485.) A description is given of the new limestone crushing and screening plant at the Beecroft Quarry, near Settle, Yorkshire. —R. A. R.

The Dithionate Process for Recovery of Manganese from Low-grade Ores. S. F. Ravitz, W. F. Wyman, A. E. Back, and K. E. Tame. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2064: Metals Technology, 1946, vol. 13, Sept.). An account is given of the development of a process for extracting manganese from low-grade oxide ores. The manganese ore is leached with dilute sulphur-dioxide gas in the presence of calcium dithionate solution, the manganese being recovered from solution by precipitation with slaked lime and then nodulized or sintered. More than 90% of the manganese was recovered from ores treated in a continuous semi-pilot plant, giving a final product containing more than 60% of manganese—C. O.

FUEL—PREPARATION, PROPERTIES, AND USES

The Combustion of Fuels. A. C. Egerton. (North-East Coast Institution of Engineers and Shipbuilders, 1946, Dec., Advance Copy). A brief survey is made of the sources of energy at the disposal of man, and of the phenomena of combustion of solids, liquids, and gases with special reference to coal, hydrocarbons, hydrogen, and the limits of combustion and flame propagation.—C. O.

The Burning of Gaseous Fuels. L. T. Minchin. (Coke and Smokeless Fuel Age, 1946, vol. 8, Sept., pp. 189–190; Oct., pp. 227–229; Nov., pp. 247–249). The combustion of gases in aerated and non-aerated flames is discussed, with reference to the theory of the mixing of gas and air streams. The third part of the article contains illustrations and descriptions of recent developments in burner design.—C. O.

Fuel Efficiency in an Integrated Iron and Steel Plant. C. Moody. (Cleveland Institution of Engineers: Iron and Coal Trades Review, 1946, vol. 153, Nov. 15, pp. 881–883). A review is made of the possibilities of further fuel economy by the utilization of blast-furnace gas in furnace practice, ingot heating, soaking pits, and gas engines.—C. O.

German Use of Low-Grade Fuel with Rich Oxygen. D. D. Howat. (Chemical Age, 1946, vol.

55, Nov. 30, pp. 661–669). A survey is made of the developments in the use of low-grade fuels in Germany made possible by the introduction of the Linde-Frankl process for the production of oxygen. Much of the information in the article, mainly on the production of water gas by the Winkler and Thyssen-Galocsy processes, is based on information given in British and Combined Intelligence Objectives Sub-Committee Reports. —C. O.

Super Cleaning of Coal. E. T. Wilkins. (Society of Chemical Industry: Iron and Coal Trades Review, 1946, vol. 153, Dec. 13, pp. 1103–1105). The need for producing coal with an ash content of 1% or less for the preparation of carbon electrodes and brushes, and for use in Diesel engines is discussed briefly. Three types of "super" cleaning process are referred to, the heavy-liquid, exemplified in the "laminar flow" or Vogel washer, the cyclone washer, and the electrostatic, such as the Johnson process used in America.—C. O.

Experiments on Coking Practice—The Effect of Type of Oven upon the Properties of the Coke. (Department of Scientific and Industrial Research, 1946: H.M. Stationery Office). The results of experiments carried out in 1938–39 under the guidance of the Coke-Ovens Committee of the Department of Scientific and Industrial Research are presented. The yields and properties of the cokes obtained from South Yorkshire, Durham, and South Wales coals in several types of oven are reported. The following trends were noted: (1) Increased rate of coking appears to cause a decrease in the size of coke; (2) the shatter strength of coke appears to decrease with increase in the rate of coking; (3) no general relationship appears to exist between the resistance of coke to abrasion and either rate of cooling or width of oven; (4) no general relationship has been determined between the reactivity of coke and the conditions of coking; (5) there are indications that the pores of coke produced in a 12-in. oven at a slow coking rate are larger than those in coke made from the same coal in a 16-in. oven at a faster coking rate.—C. O.

Experiments in Coking Practice. (Coke and Smokeless-Fuel Age, 1946, vol. 8, Oct., pp. 224–226). This article is a critical commentary on the report "Experiments on Coking Practice" (*see* preceding abstract). In spite of a great deal of elaborate and painstaking work the results do little more than confirm existing knowledge, mainly because too many variables were allowed to operate at the same time, and this prevented useful conclusions from being drawn. It is suggested that the problems awaiting solution should be studied before further research is undertaken.—R. A. R.

Experience with a Hungarian Low-Temperature Carbonization Plant. A. Romwalter. (Royal Hungarian Palatine-Joseph University, Publications of theDepartment of Mining and Metallurgy, 1941, vol. 13, pp. 46–58). The technical and economic aspects of the low-temperature carbonization plant of Salgótarján Coal Mining Ltd., at Dorog, are considered.—R. A. R.

An Automatic Gas Producer. (Overseas Engineer, 1947, Jan., pp. 183–185). A description is given of the design and operation of a small, entirely automatic gas producer developed in Great Britain during the war. Notable features of the small and compact plant are the mechanism controlling the fuel level, the thermostatically controlled ash-extraction worm, the ease of assembly of the standardized parts, and the high output of 25 therms/hr. Almost any solid fuel between the limits of charcoal and a heavily caking bituminous coal may be used, coke giving a gas of gross calorific value of 130 B.Th.U./cu. ft., and a non-caking bituminous coal one of 150 B.Th.U./cu. ft.—O. C.

Utilization of Producer Gas in Industrial Furnaces. D. B. Hendryx. (Transactions of the American Society of Mechanical Engineers, 1946, vol. 68, Nov., pp. 877–882). The economics, equipment, and methods of generating producer gas for industrial uses are discussed at length.—C. O.

TEMPERATURE MEASUREMENT AND CONTROL

A Photographic Investigation of the Brightness Temperatures of Liquid Steel Streams. J. A. Hall. (Journal of the Iron and Steel Institute, 1947, vol. 155, Jan., pp. 55–85). Cinematograph films have been taken of a range of steel streams from tapping and casting operations under such conditions as to enable an accurate density-temperature calibration of the film to be made, so that variations in brightness temperature of the steel surface can be investigated in relation to both time and space.

The effects of surface contamination and of the formation of partial black-body enclosures in the surface are discussed. The latter is found to be the most frequent cause of abnormally bright areas in the field of view. It is shown by laboratory experiment that an optical-pyrometer observer sighting on a flickering field, or on the duller parts of a non-uniform field, is likely to obtain a temperature value some 10° C. higher than that corresponding to the mean brightness of the part of the field which is being observed.

This figure is in general agreement with the results obtained on tapping streams. With casting streams, good agreement has been obtained between the optical pyrometer reading and the average photographic value of the brightness of a selected spot on the stream over a period of about a second. Under these conditions, about five out of six optical readings lie within $\pm$ 20° C. of the mean photographic value, whilst with tapping streams a similar proportion lies between 15° C. below and 35° C. above this figure.

It is shown that the use of a pyrometer employing a larger field of view than the normal disappearing-filament pyrometer (such, for example, as a photo-cell pyrometer) is not likely to introduce very serious differences. The reading will tend to be higher than that of the optical pyrometer, but the difference does not exceed 5° C. or 10° C. The variations in surface brightness have been explored by plotting frequency curves of the distribution of brightness from a large number of spots on a single frame. In this manner it has been found possible to isolate the brightness corresponding to a clear metal surface with considerable accuracy. In the future programme of work it is proposed to take certain additional precautions which have already suggested themselves in order to secure higher accuracy (especially to secure greater uniformity of exposure between successive frames of the film), and then to use the frequency-curve technique in order to make emissivity determinations on a range of steels, the true temperatures being determined by the "temperature-ring" thermocouple technique of Oliver and Land.

A Symposium on the Contamination of Platinum Thermocouples. The Liquid Steel Temperature Sub-Committee. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 213–234). The papers constituting this symposium report work carried out by the Liquid Steel Temperature Sub-Committee before it and the Foundry Steel Temperature Sub-Committee were together reconstituted in April, 1946, to form the Pyrometry Sub-Committee of the Steelmaking Division of the British Iron and Steel Research Association. Abstracts of the papers follow.—R. A. R.

An Investigation of the Embrittlement of Platinum-Rhodium Wire in the Heads of Liquid-Steel Pyrometers. T. Land. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 214–215). The embrittlement of platinum thermocouples in liquid-steel pyrometers has been investigated experimentally by exposing new thermocouple wires to different combinations of the materials in proximity to the wire in the pyrometer. It is concluded that oil or other carbonaceous materials in the thermocouple tubes are the cause of the embrittlement.

Fracture of Platinum and Platinum–13%-Rhodium Wires Used in the Immersion Thermocouple. L. Reeve and A. Howard. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 216–220). A test is described which simulates the intercrystalline attack on platinum and platinum–13%-rhodium thermocouple wires by heating a short length in a graphite block in the presence of a small quantity of lubricating oil, the wire being placed in a silica insulator. The test has been standardized at 1400° C. for 15 min., using one drop (0·05 c.c.) of Shell B. 6 oil, which contains 0·2% of sulphur. It is shown that attack is less marked on fully annealed or fully cold-worked wire than on wire which has been only partially annealed. Modified oil tests are described which confirm the conclusions of Chaston and his co-workers (described elsewhere in this symposium) that silica and sulphur are essential constituents of the attacking medium. Spectrographic examinations of attacked wires are reported, which show that copper, silicon, manganese, nickel, and iron (in decreasing order of amount) are present, particularly in the areas of maximum contamination. Thorough baking at a dull-red heat of the iron pipes used for carrying the thermocouple wires, with a current of air drawn through them, results in almost complete freedom from this type of intercrystalline failure in practice.

An X-ray Investigation of the Embrittlement of Platinum and Platinum–Rhodium Wires. H. J. Goldschmidt and T. Land. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 221–226). Platinum and platinum–rhodium thermocouple wires contaminated and embrittled in service or by experimental exposure to oil and graphite in the presence of silica, have been examined by the X-ray diffraction method. Three phases additional to the matrix were found, two of which were identified as silicides. The more severe embrittlement of the platinum–rhodium wire is explained by the formation of a phase aggregate different from that in the platinum wire. An additional observation on a platinum–rhodium wire of the point of brittle fracture in service is the segregation from the matrix, which itself is rhodium-enriched, of a finely crystalline platinum-black type of phase free from rhodium. An incidental outcome of this work is the suggested determination of the rhodium in thermocouple wires by the X-ray diffraction method.

Contamination and Failure of Rare-Metal Thermocouples. D. Manterfield. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 227–229). During an investigation into the solidification and cooling of steel ingots, failure of thermocouples occurred due to serious contamination of the couple wires. Subsequent visual, X-ray, microscopic, and spectrographic examination showed this failure to be due to the reduction of silica to silicon and the alloying of this silicon with platinum to form a new phase of platinum silicide.

Embrittlement of Platinum/Platinum–Rhodium Thermocouples. J. C. Chaston, R. A. Edwards, and F. M. Lever. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 229–231). The embrittlement of platinum thermocouple wires heated in various environments has been studied. Platinum is not embrittled by heating in contact with carbon, hydrocarbon vapours, or sulphur alone. It is shown, however, that if platinum is heated in (though not necessarily in contact with) a silicious refractory enclosure in the presence of small amounts of sulphur and carbon (as is provided, for instance, by traces of oil), embrittlement

may occur. It is suggested that in these conditions the volatile compound SiS_2 may be formed and that this attacks the platinum and forms platinum silicides at the crystal boundaries.

An Examination of the Microstructure of Contaminated and Embrittled Platinum and Platinum-Rhodium Wires. R. C. Jewell. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 231–234). Three cases of embrittlement of platinum/platinum–rhodium thermocouples have been examined microscopically. The contamination had been caused under quite different conditions in each of the three cases. In the first case, different silicides were detected in the pure platinum and the platinum–13%-rhodium wires. Severe contamination by silicon was the cause of failure, probably due to the formation of a fusible eutectic of the silicide phase and the pure platinum, and possibly also of the platinum–rhodium, aided by the brittleness of the silicide phase in the case of the platinum–rhodium wire. In the second case, intercrystalline attack was clearly revealed. The constitution of the new phase could not be determined. It was, however, clearly different from either of the silicides examined in Case 1. In Case 3 the same unidentified phase was again found.

Factors Affecting the Reproducibility of Brightness of Tungsten Strip for Pyrometer Standardization. C. R. Barber. (Journal of Scientific Instruments, 1946, vol. 23, Oct., pp. 238–242). A description is given of a quantitative examination of the effect of experimental conditions on the apparent brightness of tungsten strip lamps when used for pyrometer standardization. Appreciable differences in brightness may occur as a result of temperature gradients in the lamp filament, and of ambient temperature changes, unless certain precautions are taken. Consistent lamp polarity must be maintained because of the Thompson effect. The most satisfactory conditions for minimizing the effects of reflections from the glass envelope are shown. Cooling by convection in gas-filled lamps requires the positioning of the filament relative to the vertical to be exact.—C. O.

A Simple Automatic Furnace Control. E. L. Yates. (Journal of Scientific Instruments, 1946, vol. 23, Oct., pp. 229–231). A description is given of a furnace temperature control in which a platinum resistance thermometer forms one arm of an A.C. bridge. The out-of-balance potential is amplified, limited, and applied to the grid of an output pentode, the auxiliary grid of which is fed from an A.C. supply. The A.C. anode current of the pentode is used to operate a relay, which controls the furnace current. The instrument is stable, portable, and inexpensive. The temperature of the furnace can be controlled to within less than $\pm 0\cdot5°$ C. over the temperature range 0–1000° C. A simple method of slowly increasing or reducing the furnace temperature with time is described.—C. O.

REFRACTORY MATERIALS

Heat Treatment of Refractory Materials. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 831: H.M. Stationery Office). German methods of manufacturing basic refractory bricks, with particular reference to the design and operation of high-temperature kilns, are described.—R. A. R.

The Influence of the Glassy Bond on Some Properties of Silica Refractories. E. H. M. Badger, W. Lewcock, and J. H. Wylde. (Transactions of the British Ceramic Society, 1946, vol. 45, Aug., pp. 269–282.) A method of removing the glassy bond from silica refractories, using hydrofluoboric acid, is described. Deglassed silica, bonded with 1% of sodium tungstate, was found to have an increased coefficient of thermal expansion, most marked in the cristobalite range, but the absence of glass reduced the refractive index in the tridymite and cristobalite regions.—C. O.

The Behaviour of Refractory Materials under Stress at High Temperature. Part V. F. H. Clews, H. M. Richardson, and A. T. Green. (Transactions of the British Ceramic Society, 1946, vol. 45, July, pp. 255–268). Continued from previous articles. (*See* Journ. I. and S. I., 1946, No. II, p. 104 A). The previous parts are reviewed, and the results of further experiments are presented. Non-recoverable deformation is shown not to be of a simple viscous nature, as the rate is not proportional to the stress. From experiments on fireclay and porcelain materials in compression at 1250–1400° C. it is evident that the rate of deformation increases with the load. Proposed formulæ are discussed briefly.—C. O.

The Action of Carbon Monoxide on Refractory Materials. Part VII. Some Aspects of the Catalytic Action of the Ferruginous Glass of Iron Spots. F. H. Clews, F. Ball, and A. T. Green. (Transactions of the British Ceramic Society, 1946, vol. 45, July, pp. 251–255). Investigations are described in which samples of ferrous aluminosilicate glass, prepared in the laboratory, were subjected to the action of carbon monoxide at 450° C., (*a*) directly; (*b*) after heating in carbon monoxide or hydrogen at 900° C.; and (*c*) after heating in oxygen at 900° C. The glass was almost inert in case (*a*), but caused the decomposition of the gas in cases (*b*) and (*c*). The results are used to explain the behaviour of firebricks in service on the assumption that after a hard kiln firing, the constitution of the iron spots resembles that of the glass used in the experiments.—C. O.

Refractories—The Limiting Factor in Open-Hearth Furnace Design. J. H. Chesters. (Transactions of the British Ceramic Society, 1946, vol. 45, Aug., pp. 289–300). The limitations imposed upon the design of open-hearth furnaces by the lack of refractory materials with the requisite properties are discussed in detail. Roof and port bricks present the most urgent problems, the former because of their limiting temperature of 1680° C., which controls the input of heat to the bath, and the latter because of the serious loss of " high-temperature " heat resulting from the necessary water-cooling. The " All-Basic " type of lining has led to longer furnace life, faster melting, and a reduction in the bulk of the slag and the difficulty of cleaning pockets, but it has the disadvantages of high initial cost and greater weight. For furnaces with fixed roofs melting cold pig, bricks having greater durability than chrome-magnesite are required. Pure magnesia, alumina, and zircon offer considerable promise.—C. O.

First Report of the Converter Refractories Sub-Committee of the Steel Castings Division. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 235–269). This Report contains a factual record of the results obtained to date by the Moulds Committee of the Steel Casting Division of the British Iron and Steel Research Association. It is in five parts. The first is an outline of general current practice ; the second covers the properties of converter lining materials ; the third deals with studies of drying and preheating, and silica brick linings ; the fourth and fifth contain a summary and the acknowledgements respectively. The conclusions include the following : (1) Large vessels are more economical in the consumption of refractories than small ones ; (2) current practice relates the size of the converter to the production of the steel required per day being available from about 15 heats ; (3) monolithic linings are preferred for small vessels, with a gradual change over to silica brick for the largest vessels ; (4) improvement in lining life is obtained by charging sand into the converter before the metal, particularly when low-silicon iron is charged ; (5) the silicon content of monolithic lining materials should be over 90%, and the clay constituent should be sufficiently fine to develop adequate plasticity when present in relatively small amounts ; and (6) the relative merits of monolithic and brick linings can only be assessed by carefully controlled experiments in the same plant.

Refractories for Ovens, Furnaces and Boilers. (Coke and Smokeless-Fuel Age, 1946, vol. 8, Aug., pp. 166–168 ; Sept., pp. 196–199, 203 ; Oct., pp. 216–217, 229). The characteristics of refractory materials, the methods of manufacturing them, and their suitability for various high-temperature purposes are outlined.—R. A. R.

BLAST-FURNACE PRACTICE AND THE PRODUCTION OF PIG IRON

The Development of the Blast-Furnace Profile. E. Cotel. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1941, vol. 13, pp. 3–10). The development of the blast-furnace profile from about 1800 is traced and the tendency towards a cylindrical shape is noted. The advantages of a shaft which widens slightly towards the top are pointed out.—R. A. R.

No. 1 Blast Furnace at the Margam I. Works of Guest Keen Baldwins' Iron and Steel Co. Ltd. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 289–290). A detailed description is given of the new blast-furnace at Margam which was blown-in on 8th August, 1946. The furnace has a hearth 21 ft. 6 in. in dia. and a rated capacity of 5,000 tons/week.

The Engineering of Blast-Furnace Stoves. (Freyn Engineering Company, 1946, Pamphlet). The theoretical basis of the design of blast-furnace stoves is simply explained, and the advantages of special hexagon checker brick with round flues are enumerated. The design and insulation of stoves, and the calculation of the size and number required in particular cases are also briefly referred to.—C. O.

Blast-Furnace Practice. (Steel, 1946, vol. 119, Sept. 23, Supplement). The results of American experience in blast-furnace operation are reviewed. The majority of operators favour having more than 3 grains of moisture per cu. ft. of blast. Working with a high top pressure is not advocated. Experiments with oxygen-enriched blast are proceeding.—R. A. R.

Aluminium Blast Furnace Tuyeres. R. Guillemot. (Revue de l'Aluminium : Iron Age, 1946, vol. 158, Oct. 10, pp. 68–70). Experience with aluminium blast-furnace tuyeres in Japan, Germany, England, and France since 1920 is reviewed briefly. They have been used successfully in the first three countries since 1927, but have only been adopted in France during the last four years. The chief advantage of aluminium over copper is its lightness, which can give a saving of 25 per cent. in handling time during replacement of tuyeres. A brief description of the casting of aluminium tuyeres is appended.—C. O.

German Iron Ores Yield Vanadium. R. P. Fischer. (American Institute of Mining and Metallurgical Engineers, Technical Publication

No. 2070: Metals Technology, 1946, vol. 13, Sept.). A brief account is given of methods used in Germany during the war for the extraction of vanadium from blast-furnace slags. (*See* British Intelligence Objectives Sub-Committee Report No. 23 : Journ. I. and S. I., 1946, No. II., p. 70 A). —C. O.

TREATMENT AND USE OF SLAGS

Utilization of Blast-Furnace Slag in Germany. (British Intelligence Objectives Sub-Committee, 1946, F.I.A.T. Final Report No. 823: H.M. Stationery Office). A review of German methods of utilizing blast-furnace slag is presented. Between one-half and two-thirds of the total production of this slag has been utilized and producers have been very successful in developing cements containing a high proportion of slag ground with Portland cement clinker.—R. A. R.

Mineral Wool Plant, Gelsenkirchener Eisenwerk. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 894: H.M. Stationery Office). Two furnaces each producing about 8 tons of slag wool per day and the process of blowing the wool are described, and details are given of the manufacture of slag-wool rope 30 to 50 mm. in dia., which is a good substitute for asbestos rope. —R. A. R.

The German Mineral Wool and Heat Insulation Industries. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 916: H.M. Stationery Office). This is a report on the manufacture and use in Germany of slag wool, rock wool, and glass wool or fibre. It was concluded that the German slagwool industry had made little or no progress since 1938, and no new or specialized applications were revealed.—R. A. R.

PRODUCTION OF STEEL

Practical Interpretation of Theoretical Steel Making for the Benefit of Management and Operator. Part IV. C. F. Christopher. (Blast Furnace and Steel Plant, 1946, vol. 34, Sept., pp. 1123–1138.) Continuation of a series of articles (*see* Journ. I. and S. I., 1946, No. II., p. 132 A). The stresses set up as a steel ingot solidifies in a mould are considered, and from this the optimum shape of mould which will relieve the stresses and reduce defects is deduced.—R. A. R.

Oxygen in Liquid Open-Hearth Steel—Oxygen during the Refining Period. T. E. Brower and B. M. Larsen. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2035: Metals Technology, 1946, vol. 13, Sept.). The results are reported of a study of the oxygen content of liquid steels in the furnace and the activity of the oxygen in their slags during periods when other factors were relatively constant, usually towards the end of the refining stage. The oxygen content of the metal is independent of the slag composition over the range commonly used in acid and basic furnaces. Its normal variations in excess of the amount corresponding to equilibrium with carbon and carbon monoxide at atmospheric pressure are largely independent of any of the measured quantities such as carbon drop, iron-oxide content, or viscosity, and seem to be mainly caused by differences in condition of the hearth surface affecting the ease of bubble formation.—C. O.

Application of Slag Control and an Investigation of Basic Open-Hearth Furnace Slags. T. Fairley. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 161–171). A method of slag control for fixed basic open-hearth furnaces which is based on a combination of existing practices is described, and the results are given of a statistical survey of the chemical compositions of the melting and tapping slags of 250 casts made by the application of this method. Graphs correlating the values of slag basicities as determined by different formulæ are included.

The Determination of the Equilibrium Constant of the Reaction between Molten Iron and Hydrogen Sulphide. J. White and H. Skelly. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 201–212). Determinations of the equilibrium constant of this reaction at temperatures above the melting point of iron have previously been made by Maurer and Bischof and by Chipman and Ta Li, the values obtained by the former workers being approximately 100 times greater than those obtained by the latter.

Attempts to determine the value of the constant were made by two different methods, *viz.* (1) by the use of a high-temperature balance and (2), in a modified apparatus in which four small beads of metal were subjected simultaneously to the action of the gases in a reaction chamber small enough to be contained entirely in the hot zone of the furnace. The first method proved unreliable, owing to thermal diffusion of the gases and excessive sulphur deposition, and the results obtained by the second method are considered the more accurate. The values obtained are, on the whole, somewhat lower than those by Chipman and Ta Li. It is suggested that the true values may be still lower, as it was found that considerable amounts of silicon were picked up by the metal samples during the runs, thus apparently raising the value of the constant. There was evidence that considerable loss of silicon occurred in the H_2S–H_2 atmosphere from the refractories

used in the hot zone of the furnace, this apparently being due to the formation of a volatile silicon compound.

Blast-Furnace Gas and Oxygen-Enriched Air for the Open-Hearth Furnace. G. Bulle. (Iron and Steel Institute, 1946, Translation Series No. 294). This is an English translation of a paper which was published in Zeitschrift des Vereines deutscher Ingenieure, 1940, vol. 84, Sept. 28, pp. 740–741. (*See* Journ. I. and S. I., 1946, No. I., p. 37A).—D. R. S.

The Development of the Electric Steel Furnace during the World War 1939-1945. E. Sunstrom. (Jernkontorets Annaler, 1946, vol. 130, No. 10, pp. 477–549). (In Swedish). In England and the United States electric steel comprised 5–7% of the total tonnage produced in 1943–45; in Sweden the corresponding figure was about 30%. Swedish and foreign, especially American, electric-furnace practices are compared. American magnesite furnace bottoms had a considerably longer life than Swedish tar-mixed burned dolomite bottoms. The use of steel-clad magnesite or chrome-magnesite bricks for furnace walls is compared with the Swedish practice of ramming the walls with the same tar-dolomite mixture which is used for bottoms. The area of a self-baking Söderberg electrode is about $2\frac{1}{2}$ times the area of the ordinary graphite electrode. The advantages of basket-charging are emphasized. The metallurgy of the basic electric-furnace process is considered. Two American practices and a Swedish method of making stainless steel are described. These are: (*a*) Shortening the refining time by reboiling the bath at the end of the oxidation period; (*b*) washing the refined basic steel with its own slag by reladling the contents of the tapping ladle into a second pouring ladle; and (*c*) making stainless steel by mixing a highly alloyed heat with a low-carbon virgin metal heat. Finally, the design of high-frequency furnaces, lining problems, and some economic aspects of electric-furnace manufacture are dealt with.—R. A. R.

A Modern Electric Furnace Installation. F. W. Brooke and H. K. Georgius. (Iron and Steel Engineer, 1946, vol. 23, Oct., pp. 57–66). A steelplant built at Homestead, Pennsylvania, in 1943, is described and illustrated in detail. The melting shop is designed to take two tilting electric-arc furnaces of 55–75 tons capacity which obtain their power from a 10,000 kV.A. transformer. The furnaces are of the swing-roof type, and a specially designed charging bucket enables the complete charge to be admitted in one operation, reducing the time between tapping and switching on the power for the next heat to an average of 12 min. The shell of each furnace is a single steel casting, of 17 ft. dia.; very extensive water-cooling is employed. The electrode regulators are of the rotary variable-voltage type, which give less mechanical wear and tear and have lower maintenance costs than balanced-beam regulators. —C. O.

The Rohn Low-Frequency Induction Furnace. (British Intelligence Objectives Sub-Committee, 1946, F.I.A.T. Final Report No. 419: H.M. Stationery Office). A description is given of the Rohn low-frequency furnace installed by Messrs. Krupp during the war. It has a hemispherical bath of $1\frac{1}{2}$ tons capacity. It was wired for 2800 kVA., 220–240 V., 50 cycles, 3-phase A.C. This shape of bath enabled even solid charges to be melted. Further improvements to the refractory lining are needed to make it a competitor of high-frequency furnaces.—R. A. R.

German Ingot Moulds for the Casting of Steel Ingots. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 685: H.M. Stationery Office). This report is divided into two main sections, one on the German methods of manufacturing ingot moulds and the other on the treatment and performance of the moulds in the melting shops. At the Ruhrstahl Henrichshütte at Hattingen, the moulds for the ingot moulds were made with silica sand bonded with Portland cement. This method reduced considerably the equipment required, and the quality of the product was better than that of moulds from other German works. Brief particulars are given of certain ingot-mould maintenance practices common in Germany but not well known in England. These include (*a*) the repair of moulds by planting, chipping, and grinding; (*b*) fast stripping operations; and (*c*) the extensive use of a special mould lacquer in preference to tar.—R. A. R.

The Origin of Silicate Inclusions in Basic Electric Arc Furnace Steel of Higher Carbon Contents. A. Hultgren. (Iron and Steel Institute, 1947, Translation Series, No. 298). This is an English translation of a paper which was published in Jernkontorets Annaler, 1945, vol. 129, No. 11, pp. 633–671. (*See* Journ. I. and S. I., 1946, No. I., p. 42 A).—D. R. S.

The Theory of Segregation in Ingots. J. A. Veró. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1941, vol. 13, pp. 162–185). The solidification of ingots is described in detail and the phenomena are related to the equilibrium diagram. It is shown that no segregation can be caused by the formation and growth of the primary crystals, even in the case of solidification in layers. Gas pressure and volume

changes are the only factors during primary crystallization which produce segregation. The pressure of liberated gases may cause normal, but not inverse, segregation. The logical explanation covering all types of segregation is that normal segregation is due to an increase in volume during primary crystallization and that inverse segregation is due to a volume contraction. Liberation of gas from the alloy means a volume increase, and therefore causes normal segregation.—R. A. R.

FOUNDRY PRACTICE

Gaseous Annealing of Whiteheart Malleable Castings. P. F. Hancock. (Foundry Trade Journal, 1946, vol. 80, Nov. 28, pp. 309–316). An account is given of the development of a process for the gaseous annealing of whiteheart malleable castings. The outstanding feature is the use of coal gas as the decarburizing agent, and the regeneration of the atmosphere by burning the effluent carbon-monoxide-rich gas with a controlled volume of air, to maintain a constant carbon-dioxide content of 5–8%. Using electric or radiant-tube-heated batch type furnaces, the annealing cycle occupies 20–45 hr., depending on the size of casting. The elimination of boxes and ore, reduction in labour requirements, shorter annealing cycle, better working conditions, and improved quality and uniformity of product are some of the advantages of this process over the pack-annealing method.—C. O.

Malleable Foundry Industry. J. R. Roxburgh. (Institute of British Foundrymen : Foundry Trade Journal, 1946, vol. 80, Nov. 21, pp. 289–291). A brief review is made of current practice in the malleable iron industry. The main advances in recent years have been the use of continuous casting, centrifugal casting, synthetic moulding sands, pulverized fuel for melting and annealing, and the development of pearlitic malleable irons. —C. O.

Some Examples of Quality and Quantity Production of Iron Castings. F. Andrew. (Institute of British Foundrymen : Foundry Trade Journal, 1946, vol. 80, Nov. 21, pp. 283–288). An illustrated description is given of the methods used in the foundry of Messrs. Ferranti, Ltd., for the production of large numbers of good quality castings. The whole of the foundry is highly mechanized, which enables a daily average of 300 to 350 castings per moulding machine to be made. This high rate of production is advantageous from the point of view of the machine shops and reduces storage problems.—C. O.

Converter Process in Steel Foundries. J. H. Hall. (Foundry, 1946, vol. 74, Mar., pp. 83, 221–224 ; Apr., pp. 107, 160–164 ; May, pp. 174, 316–319). In the first part of this series of articles the operating practice for small steel-foundry converters is described. In the second part converter design is dealt with.—R. A. R.

Introduction to the Study of Foundry Sands. E. Cambier. (L'Ingénieur, 1945, vol. 27, Dec., pp. 25–43). The significance of the data obtained in the testing of foundry sands is explained and screen analyses of a number of Belgian steel-foundry sands are given.—R. A. R.

Steel Founding—Sand Practice in the Lancs-Yorks Region. R. S. Pratt. (Iron and Steel, 1946, vol. 19, Nov. 21, pp. 641–643, 771–772). An analysis of the answers to a questionnaire on moulding-sand practice sent by the British Steel Founders' Association to its members in Lancashire and Yorkshire is presented.—C. O.

The Investigation of Cast-Iron Roll Manufacture in Germany, with Notes on the Usage in Rolling-Mill Plants. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 737 : H.M. Stationery Office).—R. A. R.

Gravity Die-Casting from the Engineer's Viewpoint. M. R. Hinchliffe. (Transactions of the Manchester Association of Engineers, 1945–46, pp. 23–47). The paper deals mainly with the die-casting of non-ferrous metals, but the Holley process for iron is also mentioned. In this process, cast-iron moulds are used ; these are lined with a highly refractory material coated with lamp black from an acetylene flame. This coating is cleaned off after every casting. The moulds are air-cooled, and are usually mechanically operated.—R. A. R.

Permanent Mold Castings Offer Production Economies. W. F. Schleicher. (Iron Age, 1946, vol. 158, Oct. 3, pp. 42–45). The economics of a process using permanent metal moulds for the repetition casting of small parts are discussed. The success of the method depends mainly upon the skill with which the mould is designed.—C. O.

Atlas of Defects in Castings. Series I. (Institute of British Foundrymen, 1946). This booklet consists of a series of 34 pages of illustrations with descriptive texts which have been prepared to assist foundrymen on the correct recognition and classification of defects in castings.—R. A. R.

Experiments with a Foundry Test for the Fluidity of Molten Steel. L. W. Sanders and C. H. Cain. (British Steel Founders' Association : Foundry Trade Journal, 1946, vol. 80, Dec. 5, pp. 339–343). A description is given of a test-piece designed for

use in the foundry in the determination of the fluidity of molten steel. Tests with converter and basic electric steel gave results reproducible within narrow limits, and showed that temperature, rather than steelmaking practice, is the controlling factor in determining fluidity, which appears to increase markedly at temperatures above 1580° C. —C. O.

Common-Sense of Engineering Radiography. V. E. Pullin. (Engineer, 1946, vol. 182, Dec. 13, pp. 536–537). The advantages and limitations of the X-ray examination of engineering structures are discussed. Radiography can only yield results of practical importance and be economically applied when used by highly skilled operators under known conditions. The rôle of X-rays in the foundry is considered to be that of assisting the foundry personnel to obtain consistently sound metal rather than for the uneconomical 100% inspection of finished castings.—C. O.

HEATING FURNACES AND SOAKING PITS

Bethlehem Axle Plant Modernized. R. E. Penrod. (Iron Age, 1946, vol. 158, Oct. 10, pp. 60–63). An account is given of recent major improvements in the axle-manufacturing facilities at the Bethlehem Steel Company's plant at Johnstown, Pennsylvania. They include a continuous rotary-hearth furnace, gas-fired pits for controlled cooling of forgings, an axle-straightener of novel design, and a number of auxiliary installations. The new buildings and improvements in the layout of the plant provide an uninterrupted flow of material from the bloom store to the despatch platform.—C. O.

The Fundamentals of Industrial Furnace Design and Operation. H. Southern. (Ministry of Fuel and Power, Fuel Efficiency Committee Conference on "Fuel and the Future," London, Oct., 1946). The author discusses, with examples, the factors which contribute towards optimum efficiency in the design and operation of industrial furnaces. —C. O.

Automatic Control of Soaking Pits. C. H. Stone. (Metallurgia, 1946, vol. 35, Nov., pp. 18–22). The requirements which must be met by soaking pits and the influence of the available fuels on their design are discussed. Automatic control of the variables, pit-gas pressure, pit temperature, and gas-air ratio can give a uniformity of ingot condition and a fuel efficiency which cannot be obtained by manual operation.—C. O.

Induction Heating for Forging Economy. J. R. Coley. (Steel Processing, 1946, vol. 32, Oct., pp. 665–668). A description is given of the application of high-frequency induction furnaces to the heating of billets for forging. The principal items in the cost of induction heating are the initial cost of the equipment and the cost of the electricity used. Among the advantages to be gained are: (1) Saving in time because of the speed of heating, *e.g.* bars $2\frac{3}{8}$ in. in dia. can be heated to 2250° F. in 28 sec.; (2) exact control of temperature; (3) cleanliness and lack of scale; and (4) the ability to heat only part of a billet.—C. O.

"Portable" Induction Heating. (Steel, 1946, vol. 119, Nov. 11, pp. 166–169, 236). A very brief account is given of an induction-heating coil, which is designed for the hardening of parts inaccessible to the conventional heating units. The coil, which in appearance resembles a portable drill, operates at a frequency of 10,000 cycles/sec. —C. O.

A Practical Furnace for Vacuum Melting. A. U. Seybolt. (Metal Progress, 1946, vol. 50, Nov., pp. 1102–1106). A detailed description is given of a small high-frequency furnace designed for melting metals *in vacuo*. The maximum capacity of the furnace is 10 lb. of metal; the lowest pressures obtainable are of the order of a few microns/H_g.—C. O.

HEAT-TREATMENT AND HEAT-TREATMENT FURNACES

A Critical Survey of Controlled Atmospheres. E. J. Funk, jun., and D. von Ludwig. (Iron Age, 1946, vol. 158, Sept. 19, pp. 42–47; Sept. 26, pp. 74–79; Oct. 3, pp. 50–54). A code for identifying heat-treatment furnace atmospheres is suggested in the first part of this series of articles. In the second and third parts the principal reactions between the components of a number of atmospheres and methods of controlling them, and the attainment of equilibrium between the steel and the heat-treatment atmosphere on the basis of Dalton's law of partial pressures are discussed.—R. A. R.

Controlled Atmospheres. I. Jenkins. (Swansea and District Metallurgical Society, 1945, Feb. 17). A survey is made of the theoretical considerations involved in the heating of metals in contact with gases. It is usually desirable to prevent gas-metal reactions during heat-treatment in order to prevent the formation of surface films. The reactions in the furnace can be controlled by controlling the atmosphere. The applications and characteristics of atmospheres obtained from ammonia, hydrocarbon gases, and charcoal are discussed and compared.—C.O.

Glycerine in Steel Processing. G. Leffingwell and M. A. Lesser. (Steel Processing, 1946, vol. 32, Sept., pp. 579–581). A brief account is given of the applications of glycerine in the steel industry, including its use in the quenching, cleaning, electrolytic polishing, and etching of steel, and the binding of core sands.—C. O.

Isothermal Quenching in Commercial Practice. H. J. Babcock. (Industrial Heating, 1946, vol. 13, Aug., pp. 1288–1302). Several examples of the successful application of isothermal quenching are described. Special reference is made to the heat-treatment of splined shafts in an electrically heated salt-bath furnace.—R. A. R.

The Interrupted Quench and Its Practical Aspects. H. E. Boyer. (American Society for Metals, Nov., 1946, Preprint No. 20). The work done on the extension of the martempering process to high-carbon alloy steels is reviewed. The higher-carbon steels show a susceptibility to cracking which is not found in other types during martempering or in high-carbon steels treated by the more conventional method of direct oil-quenching. Details are given of the study of a ball-bearing steel which gave results somewhat different from those expected from experience of martempering practice.—C. O.

FORGING, STAMPING, DRAWING, AND PRESSING

Drop Forgings. J. W. Danielsen. (Transactions of the Manchester Association of Engineers, 1945–46, pp. 201–206). Progress made in drop-forging practice during the last 20 years is reviewed. The board hammer is considered the better type for hammers up to 4000 lb., but for above that weight the air or steam hammer is advocated.—R. A. R.

Forging Die Design. J. Mueller. (Steel Processing, 1946, vol. 32, Sept., pp. 572–574). The principal steps in the process of drop-hammer forging operations are described. The forging blank is shaped in several stages. The die must be so designed that sufficient material is always available to fill the die cavity, thus preventing the formation of "shuts." An example is given of a forging-die design to illustrate the sequence of operations in making a ring-forging. The grain flow produced by forging has a considerable effect on the physical properties of a product, and a good die design must take this into account.—C. O.

Methods of Production in Germany of General Heavy Forgings and Railway Axles, Tyres and Wheels. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 699 : H.M. Stationery Office).—R. A. R.

Forging Costs Cut by Improved Die Lubricant. L. P. Pistoles. (Iron Age, 1946, vol. 158, Oct. 17, pp. 55–56). An account is given of economies effected by the use of a new die lubricant consisting of a low-viscosity volatile oil, containing a small percentage of a colloid which serves to suspend fine graphite.—C. O.

The Manufacture of Hand Sewing Needles in Germany. (British Intelligence Objectives Sub-Committee, 1946, F.I.A.T. Final Report No. 864 : H.M. Stationery Office).—R. A. R.

Measuring the Efficiency of Hydraulic Presses. H. Ulbrich. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1942, vol. 14, pp. 224–235). A factor for expressing the efficiency of hydraulic presses is developed.—R. A. R.

Spinning of Stainless Steel. (Steel Processing, 1946, vol. 32, Sept., pp. 575–578). A general account is given of the advantages and limitations of the spinning process for forming stainless steel. Strong tools, heavy equipment, and low speeds are required, and frequent annealing is necessary to avoid cracking as the result of cold work.—C. O.

Wire Drawing Machines—Past, Present and Future. N. Davidson. (Wire and Wire Products, 1946, vol. 21, Sept., pp. 676–678, 690–698). Brief descriptions are given of machines used in wire-drawing, particularly those developed since the introduction of the tungsten-carbide die in the early 1920's. The author considers that the modern tendency for higher drawing speeds will not prove satisfactory until better methods of wire coating and preparation are devised.—C. O.

ROLLING-MILL PRACTICE

The Rolling of Metals : Theory and Experiment. Part VII. Factors Influencing Rolling Load and Specific Roll Pressure with Special Reference to Thin Sheet and Strip Rolling. Part VIII. Theories of Rolling Applicable to Thin Sheet and Strip. Part IX. Calculation of Specific Roll Pressure for Strip and Sheet with Strip Tension and Work Hardening by Means of the Preceding Theories. Part X. Roll Flattening and the Calculation of Specific Roll Pressure with Flattening. Part XI. Spread during Rolling. Part XII. Theories of Rolling not Restricted to Small Contact Angles and Wide Material (Mainly Hot-Rolling). Part XIII. Torque, Deformation Work and Rolling Horse-Power. L. R. Underwood. (Sheet Metal Industries, 1946, vol. 23, Jan., pp. 67–76, 85 ; Feb., pp. 269–281, 288 ; Mar., pp. 475–489, 494 ; Apr., pp. 677–684, 696 ; May, pp. 883–892 ; June, pp. 1097–1106 ; July, pp. 1291–1306 ; Aug., pp. 1495–1510 ; Sept., pp. 1699–1712 ; Oct., pp. 1901–

1914, 1919 ; Nov., pp. 2105–2115). Continuation of a series of articles (*see* Journ. I. and S. I., 1946, No. I, p. 23A).—R. A. R.

Power Requirements for Rolling and for Mill Trains. A. Geleji. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1940, vol. 12, pp. 192–212). Formulae for the power requirements for rolling rectangular and rail sections, and for the increase in the power due to the cooling of the rolled material are developed.—R. A. R.

Calculating the Forces Arising and the Power Requirements in the Mannesmann Tube-Rolling Process. A. Geleji. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1941, vol. 13, pp. 208–223). A method of calculating the forces involved and the power required in the Mannesmann process of rolling tubes is explained. The results obtained are in good agreement with the values obtained in practice.—R. A. R.

The Theoretical Problems Involved in the Design of Rolling-Mill Stands. A. Geleji. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1941, vol. 13, pp. 224–242). Methods of making the following design calculations are given : (1) Determination of the roll pressure when rolling rectangular sections ; (2) determination of the bending moment and stresses in different roll stands ; (3) determination of the elastic deformation in side frames and their effect on the rolling ; and (4) determination of the permissible stresses and deformation in side frames.—R. A. R.

Bar Mill Housings and Equipment. J. N. Imel. (Iron and Steel Engineer, 1946, vol. 23, Oct., pp. 97–103). Detailed illustrations are given of the use of movable housings for bar mills in the plant of the Jones and Laughlin Steel Corporation, Pittsburgh. The time taken in changing rolls has been reduced considerably by ensuring that the housings, rest bars, guides, and guide boxes can be easily handled and adjusted, and with a minimum of trouble.—C. O.

A Contribution on the Determination of Forward Slip. E. Cotel. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1940, vol. 12, pp. 3–9). Rolling tests are described in which the effect of changes in the speed of rolling on the amount of forward slip was determined. The forward slip increased with increasing rolling speed, *i.e.*, with the number of revolutions/sec. of the rolls. Two formulæ are developed, one expressing the percentage of forward slip in terms of the degree of reduction and the revolutions/sec., and the other incorporating a factor (derived from Puppe's work) which corrects for the rolling temperature in the 800–1200° C. range.—R. A. R.

Determination of Widening in Rolling. E. Cotel. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1942, vol. 14, pp. 3–9). A formula for calculating the increase in the width of bars after each pass is developed. The width of an initially square bar after being reduced 60% is obtained by multiplying the initial width by tan 51°. The widths after reductions of 50%, 40%, 30%, 20%, and 10% are obtained by multiplying by the tangent of the angles 50°, 49°, 48°, 47°, and 46°, respectively. The formula is compared with that of Geuze and the author finds the latter to be not quite correct.—R. A. R.

Modern Steel Providing Methods. R. W. Graham. (Iron and Steel Engineer, 1946, vol. 22, Oct., pp. 51–56). The problem of obtaining the maximum yield of finished product from a given weight of ingot entering the rolling mill is discussed. The estimation of the size and weight of ingot required to give specified sizes of semi- or wholly-finished products is known in America by the term "providing." Such a calculation necessitates a knowledge of the characteristics of the mill, the dimensions of the finished product, and the losses which may occur during the rolling operation. Rule-of-thumb methods, such as the assumption of a standard loss in weight or a constant percentage loss of weight in rolling, are very inaccurate. A simple formula has been devised to facilitate these calculations in rolling plate or strip from slabs.—C. O.

Rolling Mills—Notes on German Practice. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 824 : H.M. Stationery Office). A review of German rolling-mill design and practice is presented ; the information given was obtained in the course of visits to rolling mills and manufacturers of mill stands and all types of auxiliary equipment.—R. A. R.

Strip Production by the Steel Company of Canada, Ltd. C. Longenecker. (Blast Furnace and Steel Plant, 1946, vol. 34, Sept., pp. 1129–1142). A comprehensive account is given of the strip mill at the Hamilton, Ontario, plant of the Steel Company of Canada, Ltd. The following units are described and illustrated : The oil- or gas-fired slab-heating furnace ; the 110-in. reversing plate mill with its auxiliaries and motors ; the 56-in. hot strip mill with its motors, lubrication, and coiling mechanism ; and the shearing, cleaning, and pickling processes.—C. O.

The Rolling of Steel for Saws. P. J. Britt. (Iron and Steel Engineer, 1946, vol. 23, Oct.,

pp. 87–93). The procedure adopted in a plant at Lockport, New York, for the rolling of steel for band, hack, circular, and cross-cut saws is described.—C. O.

Electric Drives for the Uni-Temper Mill. J. C. McCurley and J. D. Campbell. (Iron and Steel Engineer, 1946, vol. 23, Oct., pp. 76–84). An account is given of the driving equipment and electrical installations of the uni-temper mill (*See* Journ. I. and S. I., 1945, No. II, p. 10 A). The very accurate speed controls necessary on the different rolls and reels are obtained by the use of electronic-amplidyne regulators.—C. O.

A Sixty-Ton Slab Turn-Over Device. W. H. Burr. (Iron and Steel Engineer, 1946, vol. 23, Sept., pp. 100–101). An illustrated description is given of an electrically driven manipulator for turning over 60-ton slabs at a very large plate mill.—R. A. R.

MACHINERY FOR IRON AND STEEL PLANT

Shunting Locomotives for Iron and Steel Works. E. L. Diamond. (Ministry of Fuel and Power, Fuel Efficiency Committee Conference on "Fuel and the Future," London, Oct., 1946). The conditions of service and performance which influence the design of locomotives for shunting in iron and steel works are discussed, with particular reference to the use of electric and Diesel locomotives in place of the present, almost universal, steam engine.—C. O.

Selecting Traveling-Crane Equipment. R. J. Wadd and F. M. Blum. (Steel, 1946, vol. 119, Nov. 11, pp. 172–175, 224–232). A system of classification of travelling cranes according to service requirements is suggested. The factors which should influence the choice of equipment, such as motors, brakes, and controls, for each of the five classes are discussed.—C. O.

Armor Plate Steels Used in Huge Coal Stripping Dipper. (Steel, 1946, vol. 119, Nov. 18, pp. 84–86). The redesign of the bucket of a power shovel to take advantage of the new steels developed during the second world war is described. The capacity of the bucket has been increased from 35 to 40 cu. yd. while the weight has been reduced by 30,000 lb. The unit may be considered an all-welded one, since even such castings as the cutter lip and the bearing connections are welded into place.—C. O.

WELDING AND FLAME-CUTTING

A Summary of the Spot Welding of High-Tensile Carbon and Low-Alloy Steels. W. D. Doty and W. J. Childs. (Welding Journal, 1946, vol. 25, Oct., pp. 624-s–630-s). A summary is given of the optimum conditions of weld diameter, electrode size, electrode force, welding time, and tempering time for eight high-strength carbon and low-alloy steels in thicknesses between 0·018 in. and 0·125 in. Great improvements in the mechanical properties of welds can be obtained by tempering in the welding machine, particularly with steels of high hardenability and thin gauge.—C. O.

Selecting Spot-Welding Schedules for Low-Carbon Steel. J. Heuschkel. (Welding Journal, 1946, vol. 23, Oct., pp. 700-s–728-s). Investigations into the production of high-strength spot-welds in low-carbon steels up to ½ in. in thickness are described. It was found that for a given combination of strength and chemical analysis, the relationships between electrode pressure, electrode size, current, and time to give any desired values of weld size, soundness, or strength, may be defined in mathematical terms. Other conclusions were: (1) Single-impulse welds are efficient for thicknesses of up to 0·30 in., if sufficient time is available; (2) the spot-welding of plates up to 0·50 in. thick is feasible if adequate power is available; (3) for heavier plates, more strength can be obtained per pound of force and per ampere required than for thinner gauge sheets; (4) single-row, spot-welded joints can be produced in low-carbon steels which develop the full strength of the unwelded metal without the necessity for tangent or overlapping welds.—C. O.

Welding Stainless Steel. C. C. Hermann. (Materials and Methods, 1946, vol. 23, Mar., pp. 713–716). Recommended techniques for welding stainless steel are submitted and discussed.—R. A. R.

Instrument Uses Supersonic Vibrations to Determine Quality Prior to Welding. O. R. Carpenter. (Blast Furnace and Steel Plant, 1946, vol. 34, Sept., pp. 1147–1148). A brief description is given of the use of the Sperry Supersonic Reflectoscope for the estimation of the quality of steel plate before welding. Plates of over 1½ in. in thickness can easily be classified in terms of suitability for welding by viewing the reflected supersonic vibrations with an oscilloscope.—C. O.

Cast Iron Repair Welding. J. M. Diebold, J. A. Blastic, and J. A. Griffin. (American Foundryman, 1946, vol. 10, Sept., pp. 49–62). An illustrated account is given of experiments on the arc- and gas-welding of grey cast iron. The test plates were all from one heat of grey iron cast under production conditions. The filler-metals used were of grey cast iron, nickel, and monel metal. Welds were made at room temperature, with preheating and postheating at 500°, 1000°, and 1400° F. Microsections

and macrosections for hardness testing were taken. Test bars incorporating welds were broken in tension. The conclusions reached were: (1) Cast-iron filler metals for both gas- and arc-welding produce structures having two-thirds of the ultimate tensile strength of the parent metal; (2) arc-welding cast-iron is as good as gas-welding under the proper conditions, and is more rapid; (3) excessive heat-treatment before or after welding is often more harmful than no treatment at all; (4) nickel filler metal makes possible the elimination of the heat-affected zone when repairing small flaws; (5) both monel metal and nickel are satisfactory materials for repair welds, without heat-treatment of the affected parts; and (6) less than 0·27% of sulphur in cast iron is a negligible factor in welding.—C. O.

MACHINING AND MACHINABILITY

Machining Wheel and Axles for Railway Rolling Stock. (Machinery, 1946, vol. 69, Oct. 17, pp. 481–486). The rolling-mill practice at the works of Taylor Brothers & Co., Ltd., was described in a previous article (*see* Journ. I. and S. I., 1946, No. II, p. 113A). In this paper the machining of wheels and axles at the same works is described.—R. A. R.

Machining Practice on Stainless Steels. W. H. Crisp and W. Burnan. (Machinist, 1946, vol. 90, Oct., pp. 1139–1143). An account of British practice in the machining of stainless steels is given. Recommendations on the types of tool for various operations, tool face angles, grinding procedure, and cutting speeds are made.—R. A. R.

Machining Stainless Steels. E. von Hambach. (Steel, 1946, vol 119, Sept. 30, pp, 62–67; Oct. 7, pp. 116–118, 150–158; Oct. 14, pp. 112–113, 134, 137). The factors involved in the machining of stainless steel are discussed. Recommendations are made for obtaining maximum production, with detailed illustrated accounts of individual machining operations, *viz.*, drilling, tapping, reaming, turning, threading, milling, broaching, filing, and sawing. The problem of lubrication is dealt with at some length.—C. O.

Cutting Fluids in Machining Operations. L. Massey and A. A. Pollitt. (Institution of Production Engineers: Metallurgia, 1946, vol. 34, Aug., pp. 213–218; Sept., pp. 275–278). The authors review and discuss the types, properties, applications, and methods of testing cutting fluids for machining operations.—R. A. R.

German Gauge and Tool Industry. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 632: H.M. Stationery Office). Visits to a large number of German firms manufacturing gauges and tools are reported. In general, methods were still in use which were discarded in England 10 to 15 years ago. An exception was Hommelwerke, where it was claimed that slip gauges were made by a sequence of lapping operations on special machines, and that slip gauges up to 100 mm. were made without any hand operation whatsoever.—R. A. R.

Poppet Valves for Automotive and Aircraft Engines. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 525: H.M. Stationery Office). A report is presented on the materials used in Germany for making poppet valves for internal combustion engines, and on the methods of manufacture.—R. A. R.

Report on German Engineers' Tool Industry. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 787: H.M. Stationery Office).—R. A. R.

PROPERTIES AND TESTS

The Mechanical Equation of State. J. H. Hollomon. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2034: Metals Technology, 1946, vol. 13, Sept.). A suggestion that under certain conditions the stress required for flow in metals depends upon the instantaneous values of the strain, strain rate, and temperature, and not upon their past values, is discussed analytically. On the basis of this "mechanical equation of state" the qualitative nature of creep may be derived from the behaviour of metals when deformed under a varying load in simple tension.—C. O.

Anelasticity of Metals. C. Zener. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 1992: Metals Technology, 1946, vol. 13, Aug.). The literature on anelasticity in metals is reviewed and discussed. Anelasticity is defined as that property of solids in virtue of which stress and strain are not uniquely related in the elastic range. The subject has been treated mathematically in terms of stress relaxation by Thomson and Wiechert, following the fundamental principles of Boltzmann. One of the physical origins of anelasticity or stress relaxation is diffusion, either thermal, atomic, or magnetic. Another common source of anelasticiity is the relaxation of shear stress across localized regions that behave in a viscous manner with respect to shear stresses and that are surrounded by an elastic matrix. It is shown that stress

relaxation is important in connection with stress relief, creep, and small dimensional changes at room temperature, and that it is closely related to fracture. Relaxation in slip bands at room temperature and across grain boundaries under creep conditions leads to the formation of the microcracks which initiate fracture. A list of 126 references is appended.—C. O.

The Technical Mechanism of Plastic Deformation. E. Siebel. (Iron and Steel Institute, 1947, Translation Series, No. 295). This is an English translation of a paper which was published in Archiv für das Eisenhüttenwesen, 1944, vol. 13, July–Aug., pp. 13–22. (*See* Journ. I. and S. I., 1945, No. II., p. 42 A).—D. R. S.

An Investigation of the Phenomenon of Cleavage Type Fractures in Low-Alloy Structural Ship Steels. H. J. Gershenow and G. G. Luther. (Welding Journal, 1946, vol. 25, Oct., pp. 611-s–615-s). An investigation is reported into the conditions of temperature and restriction of deformation under which a normally ductile steel may break with a brittle fracture when loaded in simple tension. Tests on two types of flat bar test-pieces showed that the high degree of constraint necessary to produce brittle fractures at room temperature is progressively lowered almost to zero as the temperature is reduced.—C.O.

Calculation of Tensile Strength and Yield Point from the Chemical Composition and Cooling Rate. I. R. Kramer, P. D. Gorsuch, and D. L. Newhouse. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2067: Metals Technology, 1946, vol. 13, Sept.). Details are given of tests carried out on a series of steels to establish the relationships between tensile strength, yield point, chemical composition, and rate of cooling. "Factor curves" constructed from the results enable reasonably accurate calculations to be made of the tensile strength and yield point of steels cooled at rates between those obtained at the centre of a ½-in. plate and the centre of a 16-in. plate. The most effective combination of alloying elements to produce given tensile properties in a steel cooled at a given rate may also be calculated from these factor curves.—C. O.

A Correlation of Tensile Strength and Drafting for Soft Steel Wire. M. M. Karnowsky. (Wire and Wire Products, 1946, vol. 21, Sept., pp. 669–672). A brief account is given of a method for calculating fairly accurately the tensile strength of a metal after a known reduction in cross-section in drawing, using previously established graphs.—C. O.

Improving Engine Parts by Direct Measurement of Strain. R. G. Anderson. (S. A. E. Journal, 1946, vol. 54, Sept., pp. 466–475, 502). Interpretations are given of the results of an analysis of stress distribution in machine parts. Failures in service, which are usually attributed to material defects, are in many cases caused by faulty design. Parts generally contain sufficient metal, but it is often inefficiently distributed in relation to the stresses produced in service. Residual stresses which may be beneficial should be utilized in designs.—C. O.

Elastic After-Effects in Iron Wires from 20° to 550° C. W. A. West. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 1993: Metals Technology, 1946, vol. 13, Aug.). An account is given of experiments with very pure iron wires to determine the torsional elastic after-effects produced by applied strains. The after-effect, measured as the time of relaxation shown in cold-worked wires at temperatures between 20° and 100° C., can be correlated with temperature if a heat of activation of 21,000 cal./mol is assumed; this heat of activation can be associated with the viscosity of slip bands. Calculations from observed relaxation rates of the relaxation spectrum values for the wire in the same temperature range show them to increase slowly with temperature and to be increased by cold-work. The amount of after-effect is diminished by low temperature annealing. Wires annealed at 300–350° C., which still show some after-effect of the normal type in the range 100–200° C., reveal at higher temperatures a different type, the relaxation spectrum of which increases rapidly up to 550° C. This type of after-effect has a heat of activation of 51,000 cal./mol, is not eliminated by annealing, and is reduced by increasing grain-size. It is suggested that it is due to shear-stress relaxation along the grain boundaries.—C. O.

A Study of the Damaging Effect of Fatigue Stressing on SAE X4130 Steel. J. A. Bennett. (Journal of Research of the National Bureau of Standards, 1946, vol. 37, Oct., pp. 123–137). An investigation is reported on the effect of the fatigue stressing at above the endurance limit of notched specimens of a nickel–chromium–molybdenum steel, S.A.E. X4130. A deflection method enabled the formation of fatigue cracks to be detected. Measurements of damage, *i.e.*, reduction in endurance under another stress, were confined mainly to the stages before cracks were formed. The apparent rate of damage depends on the stress history. If the damaging stress is higher than the test stress, the damage occurs rapidly at first and then more slowly, whilst the

reverse is true if the damaging stress is lower than the test stress. Tests were also made with smooth specimens to determine the cumulative effect of stressing at more than one load. Complete S-N curves were determined for specimen after each of eight different damaging treatments (where S is the stress and N is the number of stress cycles). A method of expressing damage was developed which permitted the direct addition of the damage occurring at different stresses, and the accuracy of this was checked by experiment.—C. O.

Fatigue Life of Bearings. A. B. Jones. (Steel, 1946, vol. 119, Sept. 30, pp. 68–70, 97, 99–100). An account is given of the effect on fatigue life of changes in structure of the sub-surface of high-carbon and high-alloy steels (in the fully martensitic condition) when subjected to compressive loads. Metallographic examinations were made of a number of ball bearings after use, and of a ball race of steel S.A.E. 52100 (0·95–1·10% of carbon, 1·20–1·25% of chromium). Photomicrographs show the gradual development of troostitic areas under the surface, and the formation of spalls. From a consideration of the stresses involved in the rotation of the bearings, a theory is put forward to explain the change from martensite to sorbite. The three-dimensional stress system acting on elementary sub-surface particles produces shear stresses and strains, the energy of which is not all recovered when the load is removed. Some is converted into heat by internal friction, and this results in a tempering action at those points subjected to the greatest total stress. The ultimate effect of the continued tempering action is a reduction in hardness and strength of the steel, until it is no longer able to support the stresses. The typical "spalling" fatigue failure is caused by the elliptical distribution of the maximum shear stresses under the arc of contact of the bearing and the ball race.—C. O.

Shot Peening and Its Importance in the Spring Industry. L. J. Wieschhaus. (Wire and Wire Products, 1946, vol. 21, Sept., pp. 665–667, 701–703). An illustrated description is given of the shot peening process as applied to the improvement of the fatigue properties of springs and similar articles. The surface of the article to be treated is blasted with metal shot thrown by a stream of air or from a bladed wheel. Improvements in fatigue life of up to 1370% have been reported.—C. O.

The Effect of Peening upon Residual Welding Stresses. E. P. De Garmo, F. Jonassen, and J. L. Meriam. (Welding Journal, 1946, vol. 25, Oct., pp. 616-s–623-s). A description is given of investigations into the effect of peening upon residual stresses in a number of butt welds in steel plates. It was concluded that: (1) Hot-peening does not reduce residual stresses unless the last passes are peened; (2) the longitudinal residual stresses of welds may be reduced to less than 25,000 lb./sq. in. by peening after completion and cooling, but this procedure will not appreciably reduce the transverse compression at the ends of the welds on the transverse tension in welds under transverse restraint; (3) the latter stresses may be relieved by peening the weld and adjacent metal for at least 2 in. on either side when it has cooled almost to ambient temperature; (4) the deposition of the last passes of a weld after all stresses from previous passes have been eliminated will produce residual stresses of approximately the same magnitude as would have existed had no stress relief taken place on previous passes.—C. O.

Brinell Hardness of Gray Cast Iron—Its Relation to Other Properties. J. T. MacKenzie. (American Society for Testing Materials: Foundry, 1946, vol. 74, Oct., pp. 88–93, 191–194). This paper contains an analysis of Brinell, tensile, transverse, and impact test data relating to grey cast iron. The relation between these properties, and the influence on this relationship of (*a*) low, medium, and high phosphorus contents, and (*b*) inoculation are also examined.—R. A. R.

Test Bar Data *v.* Casting Properties. C. R. Austin. (Iron Age, 1946, vol. 158, Sept. 26, pp. 70–73). Data on the hardness and tensile strength of five types of Meehanite cast iron are presented and it is shown that data obtained from specimens 1·2 in. in dia. are not applicable to castings of greater thickness. The necessity for preparing the metal during the melting process to suit the thickness of the required casting is pointed out.—C. O.

Effect of Heat Treatment on Steel Castings. E. E. Howe. (Foundry, 1946, vol. 74, Sept., pp. 98–99, 194–206). The chemical and physical changes undergone by cast steel during heat-treatment are outlined. Test data on the tensile and hardness properties of a low-alloy nickel–chromium–molybdenum steel are presented so that the changes in these properties caused by annealing, normalizing, quenching, and tempering can be compared.—R. A. R.

Recent Swedish Investigations on the Ageing of Steel. B. D. Enlund. (Jernkontorets Annaler, 1946, vol. 130, No. 10, pp. 553–573). (In Swedish). All the mild steels examined were susceptible to ageing after straining. Rapid cooling of a steel heated in the γ-range markedly counteracts ageing embrittlement, whereas a prolonged cooling time makes the steel brittle, in some cases

immediately after straining, in other cases only after ageing. Large additions of aluminium in the steelmaking process decrease the age-hardening tendency. Steels which are brittle at room temperature as a result of straining and ageing have good impact strength at temperatures over 100° C. Age-hardened steels can be restored by annealing at 600–650° C. and cooling in air.—R. A. R.

Results of Foreign Investigations on the Ageing of Mild Steel. A. Hultgren. (Jernkontorets Annaler, 1946, vol. 130, No. 10, pp. 575–591). (In Swedish). A short review is made of the results of investigations in countries other than Sweden on the quench-ageing and strain-ageing of mild steel.—R. A. R.

Effect of Tempering on Mechanical Properties. G. K. Manning and G. P. Krumlauf. (Iron Age, 1946, vol. 158, Oct. 24, pp. 44–50; Oct. 31, pp. 50–55). A description is given of the effects of variations in heat-treatment on the mechanical properties of six alloy steels. Sets of tensile and Charpy test-pieces were tempered for 2, 8, and 32 hr. at 800°, 900°, 1000°, 1100°, and 1200° F. after being quenched in water from 1600° F. All the steels showed a good correlation between Brinell hardness and tensile properties, and those steels not susceptible to temper brittleness showed a correlation between Charpy impact strength and Brinell hardness. The degree of tempering did not affect the tensile properties except in so far as it affected the hardness. The highest impact strength in the temper-brittle steels was obtained with heating for ½ hr. followed by water quenching. —C. O.

Electronic Principles as Applied in Germany to the Testing of Materials. (British Intelligence Objectives Sub-Committee, 1946, Final Report No. 724: H.M. Stationery Office). Research institutions and industrial works in Germany have been visited, and a report is given of the interrogation of personnel concerning the use of electronics in the testing of materials. It includes a summary of work done in Stuttgart during the war on the thermodynamics of alloys, the reduction of metal oxides, electrochemistry, reactions between gases and metals, the effect of supersonics on the solidification of metals, and the effect of supersonics on the course of magnetization in nickel. The "*Ringgeber*" strain gauge devised by A. Theis is described briefly.—C. O.

The Application of the Cyclograph to Non-Destructive Inspection of Materials, in Particular, Wire Rope. O. W. Ellis. (Wire and Wire Products, 1946, vol. 21, Oct., pp. 776–783). The measurement of very small changes in the physical state of metals by the use of the Dumont Cyclograph is described. This instrument utilizes the principle that the core loss of a tuned coil surrounding a piece of metal is a function of the physical state of the metal, and that changes in the physical state will cause changes in the power loss of the core, which can be shown on a cathode-ray indicator screen. A change of state in a wire rope is considered to be anything that causes a permanent increase in stress at any point, *e.g.*, abrasion, bruising, kinking, rupture of strands, and corrosion. Local defects show up as definite variations in the pattern on the screen when a wire is passed continuously through a cyclograph coil. The instrument can be used to measure plastic deformation in fatigue and creep tests. —C. O.

The Creep of Heat-Resisting Steels at Temperatures of 800° to 1200° C. E. Siebel and G. Hahn. (Iron and Steel Institute, 1947, Translation Series, No. 297). This is an English translation of a paper which was published in Archiv für das Eisenhüttenwesen, 1944, vol. 17, Mar.-Apr., pp. 211–220. (*See* Journ. I. and S. I., 1945, No. I, p. 137 A).—D. R. S.

Increasing the Creep Limit by Cold Work. H. Zschokke. (Schweizer Archiv, 1946, vol. 12, Oct., pp. 297–304). It is known that an improvement in the creep limit can be brought about by previous cold-extension of the steel. An investigation is reported in which the effects of changes in the percentage extension and in the creep-test temperature on the creep limit of three 18/8 stainless steels were investigated. The effects of 20% reduction in section by rolling at different temperatures were also studied. The following conclusions were reached: (1) Previous cold extension lowered the creep limit at above 750° C., but at below 400° C. the creep limit was improved; (2) the creep limit reached a peak in the 400–750° C. range, and then fell sharply; (3) the maximum improvement brought about by cold extension was noted in the low-temperature creep tests; in tests at 700° C. there was no improvement; (4) there was an optimum extension for each creep-test temperature.

The following observations were made regarding bars which were reduced 20% in the last pass: (1) The creep limit increased with decreasing rolling temperature; (2) the improvement in the creep limit was greatest at low testing temperatures and it disappeared at about 650–700° C.; (3) there was no peak value of the creep limit for rolling temperatures between 1000° and 660° C., *i.e.*, the critical rolling temperature was below 660° C.—R. A. R.

Removing Dissolved Gases from Molten Metals. P. M. Hume. (Steel, 1946, vol. 119, Oct. 7, pp. 108–111, 160, 163; Oct. 14, pp. 110–112, 122, 125–126, 128). The general laws of gas-metal reactions and the principal defects caused by gases in solution in metals are discussed. The limitations of the five common degassing methods, freezing and re-melting, alloy additions, decomposition of solid fluxes, melting *in vacuo*, and melting in a hydrogen-free atmosphere, are pointed out. A detailed description is given of the "flushing" process for the removal of dissolved gas, which involves bubbling an inert hydrogen-free gas through the melt. The gases commercially available for flushing are nitrogen, argon, sulphur dioxide, chlorine, and boron trichloride. The first two are the ones generally used with steel. Advantages claimed for the method are: (1) Freedom of castings from porosity due to gas; (2) elimination of "flaking"; and (3) the removal of non-metallic inclusions by flotation.—C. O.

Special Steels—Notes on Practice at Krupp A.G., Essen, and Deutsche Edelstahlwerke A.G., Krefeld. (British Intelligence Objectives Sub-Committee, 1946, Final Report 789: H.M. Stationery Office). An account is given of visits to Krupp's works at Essen, and to the Deutsche Edelstahlwerke A.G., Krefeld. The following items of special interest to Australian steelworks were investigated: (1) Welding electrodes for heat and acid-resisting steels; (2) the resistance of special steels to acids at various temperatures; (3) permanent-magnet steels, especially the "Koerzit" alloys; (4) round-ingot practice; (5) the heat-treatment of substitutes for high-speed steels; (6) an 18/8·5/1·0 chromium–nickel–tungsten steel (known as "V2AED") for gas-turbine blades; (7) substitutes for hot and cold die steels; (8) the production of stainless steel sheets; and (9) the manufacture of hardened chromium steel rolls.—R. A. R.

Heat Resisting Steels. H. Dobkin. (Steel, 1946, vol. 119, Oct. 28, pp. 79, 106–111). A general outline is given of the fundamental metallurgy of the heat-resisting steels. The necessary properties of such a steel are a high degree of corrosion resistance, satisfactory strength, ductility, and machinability at normal temperatures, and high strength at elevated temperatures. These are properties found in austenitic steels of the 18/8 chromium–nickel type. The effects of other alloying elements, *e.g.*, silicon, tungsten, molybdenum, columbium, and titanium, on the microstructures and such specific problems as intergranular corrosion, weldability, and creep resistance, are mentioned.—C. O.

Boron in Iron and Steel. R. S. Dean and B. Silkes. (United States Bureau of Mines, 1946, Sept., Information Circular No. 7363). The literature on the influence of boron on the structure and mechanical properties of steel and cast iron is reviewed. A summary is given of reported results of the effects of additions of boron to steel made in the form of the commercial alloys, Grainal, Bortam, Carbortam, and Borosil. An outline of the available methods for determining boron in iron and steel, and a comprehensive bibliography on boron, are appended.—C. O.

Effect of Alloys in Steel on Resistance to Tempering. W. Crafts and J. L. Lamont. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2036: Metals Technology, 1946, vol. 13, Sept.). A description is given of a method of calculating the hardness and tensile strength of quenched and tempered alloy steels from their hardness in the as-quenched condition, with an accuracy of 5 Rockwell C units and 15,000 lb. per sq. in. respectively. Tempered hardness is dependent upon as-quenched hardness because alloys exert their full resistance to softening only in steels hardened above a critical level. The effects of alloying elements in resisting softening are materially different from their relative effects on hardenability, so that after the same heat-treatment, steels of equivalent hardenability may vary considerably in tempered hardness and tensile strength. The order of the alloying elements in increasing ratio of softening resistance to hardening power is: Boron, carbon, manganese, nickel, chromium, molybdenum, silicon, and vanadium.—C. O.

An Improved Falling-Ball Viscosimeter. J. Proszt. (Royal Hungarian Palatine-Joseph University, Publications of the Department of Mining and Metallurgy, 1942, vol. 14, pp. 34–39). An improved falling-ball viscosimeter, which enables temperature-viscosity curves to be constructed, is described.—R. A. R.

A Practical Method of Selecting the Correct Type of Cast Iron. K. R. Geist and W. A. Hambley. (Iron Age, 1946, vol. 158, Oct. 17, pp. 64–70; Oct. 24, pp. 58–64). Data collected in a grey-iron foundry during the last five years are presented. Graphs and photomicrographs show how correlations can be established between melting and casting histories, chemical composition, and physical properties, which should enable the correct conditions to be obtained for meeting a given specification.—C. O.

A Practical Method of Specifying Cast Irons. [.] R. Geist and W. A. Hambley. (Iron Age, [1]946, vol. 158, Oct. 31, pp. 46–49). A simple [p]ractical method is explained for selecting the [g]rade of iron suitable for meeting a given specifica[ti]on. The scheme is based on a knowledge of the [re]lations between the chemical composition of an [ir]on and the physical properties to be expected in [ca]stings of a given controlling cross-section.—[c]. o.

METALLOGRAPHY

Austenitic Grain Size. W. I. Pumphrey. [(I]ron and Steel, 1946, vol. 19, Oct., pp. 581–583). [A] review is presented of a number of etching [m]ethods for determining the grain size of the [a]ustenite at the quenching temperature in a [h]ypereutectoid chromium–molybdenum steel [q]uenched from a variety of initial conditions [r]anging from fully annealed to air-cooled from [h]igh temperatures.—c. o.

The Application of Electrolytic Polishing for [F]errous Metallography. H. J. Merchant. (Iron [a]nd Steel Institute, 1947, vol. 155, Feb., pp. 179–[1]94). Preliminary experiments on electrolytic [p]olishing were conducted to determine the effects [o]f variable conditions of electrolysis when using [J]acquet's perchloric-acid/acetic-anhydride electro[l]yte. The design of a suitable type of cell was [a]lso investigated and five different cells were [c]onstructed. Electrolytic polishing is compared [w]ith mechanical polishing on the basis of [p]rocedure and results obtained; specimens [c]ontaining the graphite phase, large amounts of [s]lag, or discontinuities, do not respond to electro[l]ytic polishing. Other potential solutions for [e]lectrolytic polishing were also investigated, and from these a list of recommended electrolytes is given. The most useful acids for metallographic purposes are perchloric acid, nitric acid, and chromic acid, diluted where possible with acetic anhydride, acetic acid, or alcohol. Electrolytes based on phosphoric acid or sulphuric acid cannot be recommended for metallographic polishing owing to their tendency to produce pitting or polish attack. Various observations made in the course of this work are then considered relative to the present theory of electrolytic polishing.

Fluoroscopic Inspection of Steels. G. C. Close. (Steel Processing, 1946, vol. 32, Aug., pp. 502–504). A brief illustrated description is given of a machine for the fluoroscopic examination of castings. The article to be inspected is suspended in the path of an X-ray beam, which causes its image to be thrown upon a screen consisting of a lucite cell, 4 in. in thickness, filled with a saturated solution of lead perchlorate. The installation is less costly, and the process is much more rapid than radiography. Castings can be easily examined from all angles. On the other hand, no permanent record is obtained, more power is required to produce visible images than to produce X-ray negatives, and the human element enters largely into the interpretation, which must be done immediately. Fluoroscopic inspection will probably supplement the X-ray method, rather than become an accepted standard.—c. o.

Microstructure of Hard-Facing Alloys. H. W. Sharp. (Welding Journal, 1946, vol. 25, Oct., pp. 936–941). The microstructures found in the four groups of alloys commonly used for hard-facing are described and illustrated. Group 1 alloys, which are iron base with less than 20% of alloying elements, mainly chromium, manganese, and silicon, with high carbon, have generally a pseudo-martensitic structure with scattered particles of chromium carbide. The iron-base alloys of group 2, which contain over 20% of elements such as chromium, manganese, tungsten, molybdenum, and nickel, have structures consisting of islands of complex chromium carbides in a matrix of iron and manganese. The alloys of cobalt, chromium, and tungsten which form the third group consist of particles of complex chromium and tungsten carbides in a cobalt solid-solution matrix. The fourth group, consisting of tungsten carbide dispersed in mild steel, has a heterogeneous structure, with particles of the carbide irregularly embedded in a mild steel matrix.—c. o.

The Microstructure of Commercial Acid-Resisting Silicon–Iron Alloys. J. E. Hurst and R. V. Riley. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 172–178). Difficulties in the preparation of metallographic specimens and in the interpretation of the microstructures of acid-resisting silicon iron have been largely overcome. The disposition of the phases in commercial irons containing between 14% and 18% of silicon has been found to agree on the whole with what would be expected from a study of the most recent iron–silicon binary diagram. The influence on the microstructure of variations in silicon content, additions of certain alloying elements, and of adventitious impurities has been studied, and photomicrographs are presented.

The Surface Tension of Metals. A. Brager and A. Schuchowitzky. (Acta Physicochimica U.R.S.S., 1946, vol. 21, No. 1, pp. 13–22). Several theories to account for the high surface tensions of molten metals are discussed. On the basis of

Sommerfeld's model of a metal, it is shown that the dispersion of a metal results in an increase in the kinetic energy of its electrons, which is the main physical cause for the high values of surface tension. An equation is derived for the calculation of the surface tension of metals, which gives the correct order of magnitude, and expresses its dependence on the electron cloud density. Surface tensions may be calculated by the method of molecular orbitals.—C. O.

Effect of Original Orientation on Orientation Changes during Recrystallization in Silicon Ferrite. C. G. Dunn. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 1990: Metals Technology, 1946, vol. 13, Aug.). The work described in this paper extends that on recrystallization in silicon ferrite (*see* Journ. I. and S. I., 1945, No. II., p. 97 A) to include five original orientations and higher percentages of cold reduction. Analyses of 351 examples of recrystallization gave a high probability for obtaining a third-order twin relationship between new and old structures. Orientation changes due to recrystallization are classified best as C_3 regardless of original orientation. With greater amounts of cold reduction, or with increasing spread in orientation of the deformed grain, there was an increase in the spread of orientations away from the C_3 Classification.—R. A. R.

Some Aspects of Crystal Recovery in Silicon Ferrite Following Plastic Strains. C. G. Dunn. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 1991: Metals Technology, 1946, vol. 13, Aug.). Investigations of the recovery of plastically deformed metals are reported. Recovery has been divided into three classes: (1) The removal of dislocations within mosaic blocks, (2) the removal of elastic strains, and (3) the removal of distortions that produce a spread in orientations over regions that are larger than 10^{-4} cm., *i.e.*, larger than mosaic blocks. The third class of recovery was studied by Laue diffraction patterns made from single crystals of silicon-ferrite sheets. This recovery can occur to the extent of almost 100%, when it produces sharp Laue spots in X-ray diffraction patterns. The amount of recovery that occurs in about 15 min. at 1100° C. seems to be sufficient to reduce the rate of growth of a new grain almost to zero.—R. A. R.

The Suppression of Pearlite in Manganese–Molybdenum Steels. C. R. Austin and J. R. Doig. (Transactions of the American Society for Metals, 1946, vol. 36, pp. 336–360). A study was made of the effects of carbon, manganese, and molybdenum on critical cooling rates in hypo-eutectoid manganese–molybdenum steels. From S-curve data and a formula developed by Grange and Kiefer (*see* Journ. I. and S. I., 1941, No. 1, vol. 143, p. 76 A), it was possible to calculate the velocity of cooling required to prevent the formation of pearlite. The influences of manganese and molybdenum on the times for isothermal transformations of pearlite at 650° C. were determined, and the results enabled an approximate formula to be derived expressing the critical cooling velocity for pearlite suppression as a function of the total content of the alloying elements.—C. O.

Anisothermal Decomposition of Austenite. J. H. Hollomon, L. D. Jaffe, and M. R. Norton. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2008: Metals Technology, 1946, vol. 13, Aug.). The available information on the relations between the isothermal and anisothermal decomposition of austenite is presented and correlated, and new experimental work on them is described. The authors discuss the concept of "additivity" in anisothermal decompositions involving only one reaction. The hypo-eutectoid ferrite and the pearlite reactions appear to be roughly additive, but the bainite reaction is not, since the residual austenitic carbon content varies. Hypo-eutectoid ferrite in small quantities nucleates and so increases the rate of the bainite reaction, but in large quantities it hinders bainite formation. Hypo-eutectoid carbide has the opposite effect. Martensite facilitates the nucleation of bainite, but pearlite does not seem to have any influence. Small amounts of hypo-eutectoid products have little effect upon the pearlite reaction, whereas moderate amounts of high-temperature bainite and martensite hasten transformations to pearlite.—C. O.

Nitrogenation of Iron by Molecular Nitrogen at High Pressures. I. Kritchevsky and N. Khazanova. (Acta Physicochimica U.R.S.S., 1946, vol. 21, No. 1, p. 187). The authors have calculated on thermodynamical grounds that the equilibrium pressure of nitrogen in the system Fe (α phase)—Fe_4 N(γ' -phase)—N, at 475° C. is equal to 2580 atm. and not 5450 atm. as obtained in a simplified calculation by P. Emmett, S. Hendricks, and S. Brunauer.—C. O.

The Boron-Oxygen Equilibrium in Liquid Iron. G. Derge. (American Institute of Mining and Metallurgical Engineers, Technical Publication No. 2004: Metals Technology, 1946, vol. 13, Aug.). A description is given of experiments designed to determine the relations between boron and oxygen in liquid iron. Melts made in

ca and magnesia crucibles under slags contain-; varying amounts of iron and boron oxides re analysed for boron and oxygen. It was found at the deoxidizing power of boron is comparable th that of silicon, and can be expressed by the nstant

$$B=\frac{1}{B_2\,(\%\text{ liquid Fe})\times O^3\,(\%\text{ liquid Fe})}=2\cdot4\times10^8$$

The amounts of boron which are of commercial terest can be introduced into steel from boron ide compounds if the amount of oxygen esent is suitably controlled.—C. O.

The Miscibility Gap in the Fe–S–C System at rbon Saturation in the 1300–2000° C. Tempera-re Range. A. Norrö and S. Lundquist. (Iron d Steel Institute, 1947, Translation Series, o. 299). This is an English translation of a per which was published in Jernkontorets nnaler, 1946, vol. 130, No. 3, pp. 118–126. *ee* Journ. I. and S. I., 1946, No. II, p. 13A).— R. S.

CORROSION

The Corrosion of Pipe Lines for Hydraulic resses Can Be Prevented by Perfecting the esign. H. Ulbrich. (Royal Hungarian Palatine-oseph University; Publications of the Depart-ent of Mining and Metallurgy, 1942, vol. 14, p. 236–255). The solubilities of oxygen and itrogen in water at different pressures and mperatures are considered and the amounts berated at different drops in pressure are pproximately derived. Corrosion in the pipe nes for hydraulic presses is shown to occur at ositions in horizontal lines after the places where pressure drop occurs. Proposals for pipe-line esign, with special reference to the position of ontrol valves, are made.—R. A. R.

Investigation and Control of Condensate Well orrosion. P. L. Menaul. (Corrosion, 1946, ol. 2, Oct., pp. 195–198). A method for deter-ining the total organic acid content in oil-well ondensate water and equipment for injecting hemicals into the water to prevent corrosion are escribed.—R. A. R.

Atmospheric Corrosion Tests on High Chromium teels. W. O. Binder and C. M. Brown. (Steel Processing, 1946, vol. 32, Sept., pp. 565–571, 94–595). Results are given of corrosion tests on ow-carbon steels containing up to 18% of hromium carried out since 1941 in marine tmospheres at Kure Beach, North Carolina, and n industrial atmospheres at New York City and Niagara Falls. The specimens, of 16-gauge sheet, were exposed at an angle of 30° to the horizontal. The Kure Beach samples were corroded uniformly n both sides. The others appeared to be more corroded on the earthward sides. Photographs and curves of weight loss against chromium content show that the amount of corrosion decreases as the chromium content increases. With 12% of chromium, the rusting is very slight, while at 18% it is practically nil. The marine atmosphere is in general less corrosive than the industrial atmosphere. Further tests were undertaken to study the effects of exposure on cold-rolled austenitic chromium–nickel and chromium–manganese–nickel steels. Since the change in weight, even after five years, was so small, the effect of corrosion was evaluated by tensile tests. Exposure to the industrial atmosphere of Niagara Falls, for five years, did not affect the strength or ductility of tensile test pieces. Spot-welded sections, similarly exposed, remained unimpaired in strength and free from permanent staining.—C. O.

Some Experiments on Corrosion of Steel by Boiling Water. A. G. Gould and U. R. Evans. (Journal of the Iron and Steel Institute, 1947, vol. 155, Feb., pp. 195–200). The behaviour of steel in boiling water is shown to be determined largely by the position of the formation of solid corrosion products. Under anaerobic conditions steel reacts at first with water, eliminating hydrogen, but a film (first probably ferrous hydroxide, then becoming magnetite) is formed over the surface, so that the attack over long periods is very slight. In the presence of oxygen the conversion of ferrous hydroxide to magnetite or rust appears to take place to some extent at a distance from the metal, so that the film is discontinuous and serious corrosion occurs. Salts tend to increase, and sodium hydroxide to diminish, the attack under aerobic (but not under anaerobic) conditions. Copper deposited on part of the steel increases the intensity of attack in short experiments but not in long ones. Oxide films formed by previously heating in air, if interrupted by a scratch-line, can greatly increase the intensity of the attack.

ANALYSIS

Semi-Quantitative Tests for Moly in Steel. W. E. Thrun and C. R. Heidbrink. (Iron Age, 1946, vol. 158, Oct. 3, p. 41). A description is given of modifications of the thiocyanate and potassium-ethylxanthate methods for detecting and roughly estimating molybdenum in steel and iron. A determination can be made in about 4 min. with reasonable accuracy for up to 0·5% of molybdenum.—C. O.

Determination of Copper in Cast Iron and Steels with Quinaldic Acid. J. F. Flagg and D. W. Vanas. (Industrial and Engineering Chemistry,

Analytical Edition, 1946, vol. 18, July, pp. 436–438). The method of A. M. Zan'ko and G. O. Butenko for the determination of copper in steel by precipitation with quinaldic acid has been investigated and extensively modified. Satisfactory results are obtained only after a re-precipitation of the copper quinaldate. The speed of the method may be increased by making a preliminary separation of the copper by internal electrolysis using an aluminium anode. The method, as applied to Bureau of Standards steels, gave an accuracy of about 2% to 3% on samples containing $0 \cdot 1\%$ or more of copper, but the accuracy was less for lower contents.—R. A. R.

Colorimetric Determination of Cobalt in Metallurgical Products with Nitroso R Salt. R. S. Young, E. T. Pinkney, and R. Dick. (Industrial and Engineering Chemistry, Analytical Edition, 1946, vol. 18, Aug. 16, pp. 474–476). A description is given of an adaptation of the nitroso-R salt method of determining cobalt, to enable small quantities of the metal present in metallurgical materials to be estimated. Results obtained compare favourably in accuracy with those given by the longer standard gravimetric procedures. Details are given for the use of the Spekker absorptiometer in the final stages, but any other means of photo-electric or visual comparison may be used.—C. O.

BOOK REVIEWS

American Iron and Steel Institute. "*Classified Provisions of Forty Collective Bargaining Agreements for Wage Earners in the Iron and Steel Industry.*" 4to, pp. viii + 737. New York, 1, N.Y., 1946 : The Institute.

This volume, prepared under the direction of the Industrial Relations Committee of the American Iron and Steel Institute, presents in classified form the provisions of forty current collective bargaining agreements for wage-earning employees in the iron and steel industry.

It has been prepared primarily for the information of members of the industry and with a view to providing a ready reference source on occasions when new agreements must be negotiated.

Bunn, C. W. "*Chemical Crystallography. An Introduction to Optical and X-Ray Methods.*" La. 8vo, pp. xii + 422. Illustrated. Oxford, 1946 : Clarendon Press. (Price 25*s*.).

This book deals with methods which have been developed for the identification of solid bodies by microscopical and X-ray methods (by determining molecular weight, size, shape, and symmetry), and for a detailed study of stereochemistry by crystallographic methods. The book includes an account of the elements of crystal morphology and optics, the interpretation of various types of X-ray diffraction photographs, and the location of atoms both by the method of trial and error and by Fourier-series methods.

Burton, E. F. and W. H. Kohl. "*The Electron Microscope. An Introduction to its Fundamental Principles and Applications.*" Second Edition. 8vo, pp. 325. Illustrated. New York, 1946 : Reinhold Publishing Corporation. (Price 30*s*.).

The text of this edition has been re-arranged and revised in many places to attain greater coherence in the presentation of the subject matter. Some material has been eliminated as being of too specialized a character for the scope of the book. The chapter on the compound magnetic electron microscope has been re-written and much new material added. The chapters on the applications of the electron microscope has also been largely re-written. A complete bibliography of the literature on electron microscopy is included.

Forrester, J. D. "*Principles of Field and Mining Geology.*" 8vo, pp. viii + 647. Illustrated. New York, 1946 : John Wiley and Sons, Inc. ; London : Chapman and Hall, Ltd. (Price $7.).

This volume, by the former Professor and Head of Geology, School of Mines, University of Idaho, has been written, not for the beginner in the study of geology, but for students already familiar with the elementary principles of the subject. Its object is primarily to acquaint students with field procedure and technique in mining geology, but parts of it will be useful also for practising geologists and civil engineers.

The first of the four parts of the book deals with the recognition and correlation of geological phenomena, including faults, cleavage, and folded structures, and it contains a very general description of the nature of some of the chief mineral deposits. Part II., entitled "General Field-Survey Practices and Procedure," gives an excellent summary of field methods, equipment, instruments and surveying tables, and is perhaps the most valuable part of the volume. Part III., contains much useful matter about underground geological mapping, sampling technique, drilling methods and application of geophysical methods of exploration. English readers will not be very interested in the section of this Part dealing with Federal mining statutes in the U.S.A. Part IV., "The Interpretation and use of Field Data," contains considerable information concerning the preparation of geological maps and sections, the interpretation of structural controls in locating ore deposition, the treatment of sample essay analyses, estimation of ore reserves, and the preparation of geological reports.

Within 647 pages it is quite impossible to deal adequately with the wide range of subjects attempted in this volume, and it is not surprising that some aspects, such as the mode of occurrence of mineral deposits and the recording of surface geology, are somewhat sketchy. Nevertheless, this book, profusely illustrated with excellent drawings, and pregnant with practical information not accessible in any other single volume, can be recommended strongly to students in mining and engineering geology and those practising in these subjects.

W. R. Jones.

Presgrave, R. "*The Dynamics of Time Study.*" Second Edition. 8vo, pp. xvii + 238. Illustrated. New York and London, 1945 : McGraw-Hill Book Co., Inc. (Price 12*s.* 6*d.*).

This book analyses the essential principles to which sound time-study technique must conform and describes practical methods for applying these principles.

Sandy, A. H. "*Cutting Tools for Engineers.*" 8vo, pp. x + 130. Illustrated. Revised Edition. London, 1946 : Crosby, Lockwood and Son, Ltd. (Price 5*s.*).

This volume is a revision of the original work published in 1939, and it should provide a concise introduction to a complex subject for engineers and metallurgists. The author's approach to his subject is essentially practical and he imparts to the reader all that is modern and good in workshop practice. Theory, which, unfortunately, lags sadly behind practice in cutting-tool studies, is not neglected, but is given all the mention it deserves and there Mr. Sandy does well to leave it.

There are twelve chapters, admirably dividing up the ground to be covered. The first four are very short and deal with general considerations, the mechanics of metal-cutting, and with tool materials. The next six chapters describe modern practice as applied to hand tools, drills, lathe tools, milling cutters, broaches, and gear-cutting tools. The two final chapters are on tool and cutter grinding and cutting lubricants.

The author writes clearly and has incorporated many line drawings which are distinctly helpful in visualizing the three-dimensional nature of most machining operations. The tables of data provided will also be useful, and assist the reader in gaining a wider view of present-day practice. The volume is well indexed and is pleasingly printed and bound for its modest price.

S. L. Roberton.

Simons, E. N. "*Saws and Sawing Machinery.*" 8vo, pp. vii + 224. Illustrated. First Edition. London, 1946 : Sir Isaac Pitman and Sons, Ltd. (Price 15*s.*).

The author, in his foreword, states that, to the best of his knowledge, this volume is the first to be published exclusively concerned with all types of saws, and as such it is bound to be a most useful work of reference. Although the book is the personal work of the author he has taken full advantage of his connection with many specialists in sawing machinery to have his text checked so that it is fully up to date and accurate, and the result is a comprehensive volume covering an important branch of hand and machine tools.

Mr. Simons covers the entire field of application of saws, the cutting of timber, metals, and stone, all types of saw being dealt with. The subject is subdivided into the various types of saw, and a consideration of the nature of the work carried out. The first chapter is brief and historical, and the second deals with the simplest and most familiar member of the saw family, the wood hand saw. Chapter III. introduces circular saws generally, and the specific types used for wood and metal are described in detail in the two succeeding chapters. Then follow chapters on inserted tooth and segmental saws, circular friction discs and diamond saws. The last three chapters cover the hacksaw, bandsaws, and contour sawing. In two appendices, the author gives a survey of safety precautions for circular-saw users, and a series of tables of interest to all who use sawing equipment. The book is well printed and is generously provided with half-tone and line illustrations which appreciably assist the text. The writing is explicit and concise, and the arrangement of the subject matter is good. It may therefore be said that the gap in informative literature on this subject has been well filled by Mr. Simons' comprehensive and authoritative book.

S. L. Roberton.

Sokolnikoff, I. S. "*Mathematical Theory of Elasticity.*" With the collaboration of R. D. Specht. First Edition. 8vo, pp. xi + 367. Illustrated. New York and London, 1946 : McGraw-Hill Book Co., Inc. (Price 22*s.* 6*d.*).

The author presents a lucid treatment of the basic theory of mechanics of deformable media. The first three chapters (entitled Analysis of Strain, Analysis of Stress, and Stress-Strain Relations) provide a foundation in the mathematical theory of elasticity. The latter portion of the book is concerned with the application of the theory to problems on extension, torsion, and flexure of isotropic cylindrical bodies and with variational methods in the theory of elasticity.

Wiren, A. R. and C. Heyel. "*Practical Management Research.*" 8vo, pp. x + 222. New York, 1945 : McGraw-Hill Book Co., Inc.; London : McGraw-Hill Publishing Co., Ltd. (Price 14*s.*).

This book is concerned with the application of research principles to general management problems as well as to specific operating functions.

Wood, V. N. "*Metallurgical Materials, Alloys and Manufacturing Processes.*" With a Foreword by J. W. Gardom. 8vo, pp. xi + 340. Illustrated. London, 1946 : Chapman and Hall, Ltd. (Price 25*s.*).

This book is intended primarily for engineering and metallurgical students. Early chapters deal with the manufacture of iron and steel, mechanical testing, and physical examination. Chapters follow on industrial control and measurement of temperature, heat-treatment and mechanical working of steel, and the properties of carbon and alloy steels. Chapters are also included on cast iron, malleable cast iron, non-ferrous metals and alloys. Finally, there are chapters on welding and galvanizing, and the use of metals and alloys during the war period.

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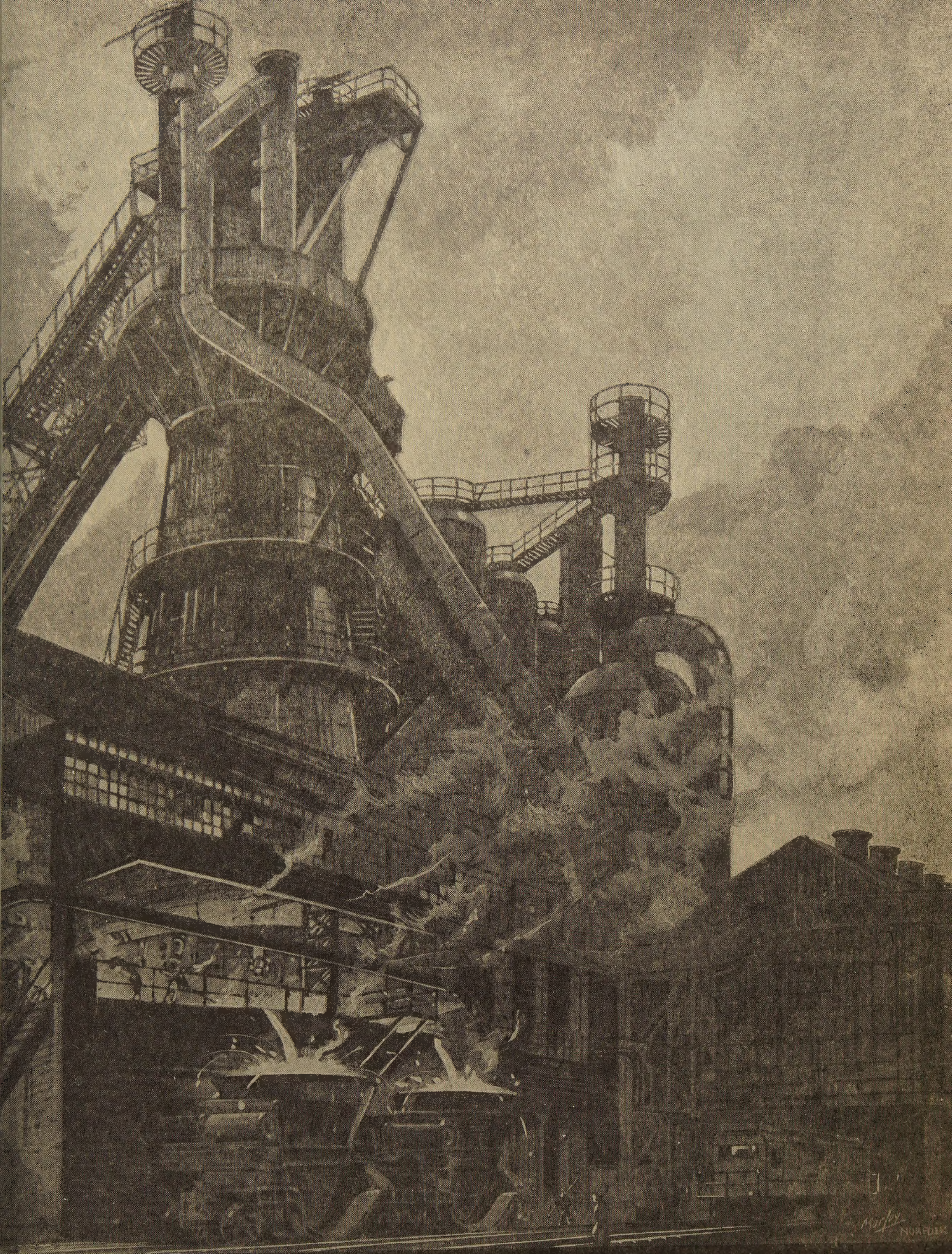

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